

# PATENT ABSTRACTS OF JAPAN

(11)Publication number : 09-120088

(43)Date of publication of application : 06.05.1997

(51)Int.Cl.

G02F 1/15  
C09K 9/02

(21)Application number : 08-219694

(71)Applicant : GENTEX CORP

(22)Date of filing : 21.08.1996

(72)Inventor : BYKER HARLAN J

(30)Priority

Priority number : 86 846354    Priority date : 31.03.1986    Priority country : US

(54) SINGLE BLOCK TYPE SELF-ERASURE SYSTEM SOLUTION PHASE ELECTROCHROMIC DEVICE AND SOLUTION USED TO THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To enable the commercial embodiment of the use of this display device by using a specific soln. as a variable transmittance medium.

SOLUTION: This soln. consists of (A) a solvent; (B) a cathode electrochromic compd. which exhibits  $\geq 2$  kinds of chemically reversible reduction waves in the voltamogram executed by using an inert electrode at room temp. in this solvent and in which the wave of the first kind entails an increase in the molecular extinction coefft. at a wavelength of a visible range; (C) an anode electrochromic compd. which exhibits  $\geq 2$  kinds of chemically reversible oxidation waves in its voltamogram and in which the wave of the first kind entails an increase in the molecular extinction coefft. at a wavelength of a visible range; (D) a current carrier electrolyte which is insert when all of the cathode compd. and the anode compd. are not ionic in the respective zero potential equil. state in this solvent. The soln. described above is used as the variable transmittance solvent in the single block self erasure system soln. phase electrochromic device.

## LEGAL STATUS

[Date of request for examination] 20.09.1996

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] 2672083

[Date of registration] 11.07.1997

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

\* NOTICES \*

Japan Patent Office is not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

## CLAIMS

[Claim(s)]

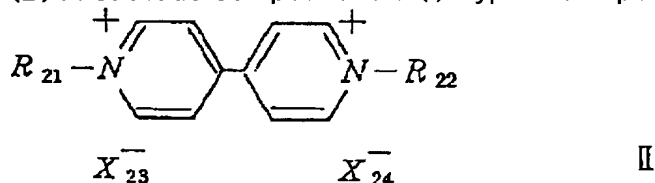
[Claim 1] (A) A solvent;

(B) At least one sort of cathode electrochromic compounds accompanied by [ in / among these reduction / in a voltamogram performed in a room temperature in the above-mentioned solvent, display at least two sorts of chemical reversible reduction waves, and / at least one sort of wavelength of a visible region ] increase of a molar extinction coefficient in the 1st thing;

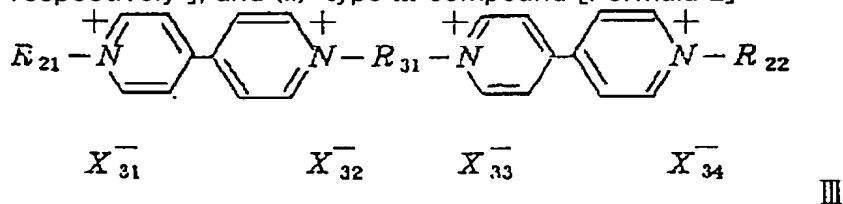
(C) Display at least two sorts of chemical reversible oxidation waves in a voltamogram performed in a room temperature in the above-mentioned solvent. The 1st thing is accompanied by increase of a molar extinction coefficient in at least one sort of wavelength of a visible region among these oxidation. When all of at least one sort of anode electrochromic compound; and (D) cathode compounds, and an anode compound are not ionicity in those zero potential equilibrium in the above-mentioned solvent A solution used as an adjustable permeability medium in a single partition mold self-elimination type solution phase electrochromic device which consists of an inactive current conveyance electrolyte.

[Claim 2] (A) A solvent is a liquid at a room temperature, and are two or more sorts of above-mentioned solvents which are liquids at propylene carbonate, gamma-butyrolactone, gamma-valerolactone, and a room temperature, or is chosen from homogeneous mixture of one or more sorts of above-mentioned solvents, and ethylene carbonate.;

(B) A cathode compound is a (i)-type II compound. [Formula 1]

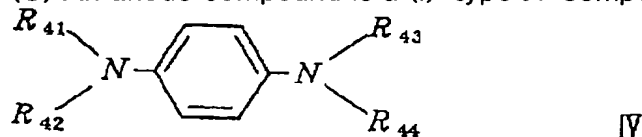


Whether R21 and R22 are the same among [type Or the alkyl group which differ and has 1-10 carbon atoms, respectively, a phenyl group (cyano \*\* the location of arbitration -- setting -- chloride, bromide, and iodide --) Or you may be replaced by the alkyl group which has 1-4 carbon atoms. and benzyl (a phenyl group -- the location of arbitration -- setting -- chloride and bromide --) It is chosen [ that you may be replaced by the alkyl group which has iodide and cyano \*\* or 1-4 carbon atoms and ], and whether; X-23 and X-24 are the same Or a difference, ]; [ which is chosen from chloride, bromide, iodide, BF<sub>4</sub>-, PF<sub>6</sub>-, AsF<sub>6</sub>-, ClO<sub>4</sub>-, and NO<sub>3</sub>-, respectively ], and (ii)-type III compound [Formula 2]

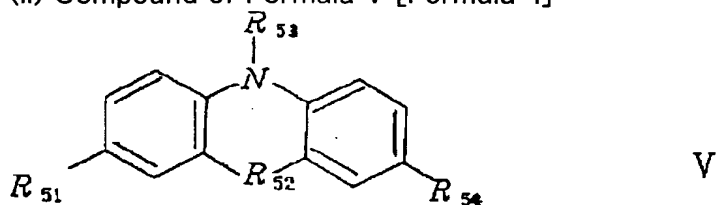


(-- or [ the inside of a formula, and / that R21 and R22 are the same ] -- or -- differing -- previously -- the compound of Formula II -- setting -- R31 -- 1- -- or [ that are the alkylene group which has ten carbon atoms, and X-31, X-32, X-33, and X-34 are the same ] -- or -- differing -- respectively -- chloride, bromide, iodide, and BF<sub>4</sub>- -- it is chosen out of 4, PF<sub>6</sub>-

(C) An anode compound is a (i)-type IV compound. [Formula 3]

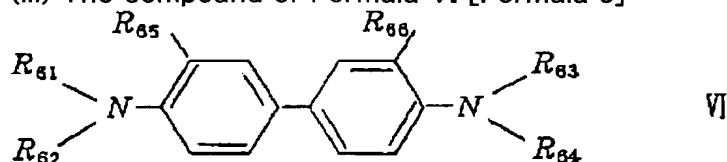


Among [type whether R41, R42, R43, and R44 are the same Or a difference, the alkyl group which has a hydrogen atom and 1-10 carbon atoms, respectively, and a phenyl group (the location of arbitration -- setting -- chloride --) You may be replaced by the alkyl group which has bromide, iodide, and cyano \*\* or 1-4 carbon atoms. And] chosen from benzyl (the phenyl group may be replaced by the alkyl group which has chloride, bromide, iodide, and cyano \*\* or 1-4 carbon atoms in the location of arbitration);  
(ii) Compound of Formula V [Formula 4]



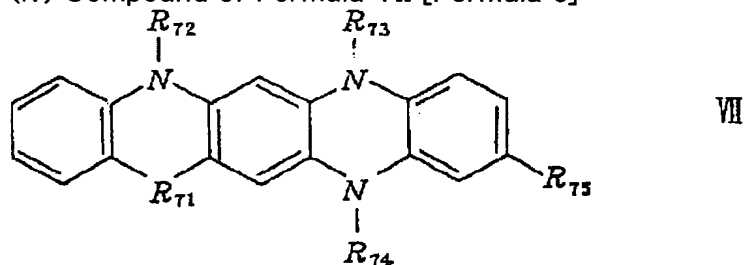
or [ the inside of [type, and / that R51 and R54 are the same ] -- or -- differing -- respectively -- a hydrogen atom and a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) It is chosen out of having 1-6 carbon atoms, respectively.;R52 An oxygen atom, Whether it is a sulfur atom or NR55, and R55 is the same as that of R53 here Or a difference, The alkyl group in which both R55 and R53 have a hydrogen atom and 1-10 carbon atoms, a phenyl group (cyano \*\* the location of arbitration -- setting -- chloride, bromide, and iodide --) Or you may be replaced by the alkyl group which has 1-4 carbon atoms. Or] chosen from benzyl (the phenyl group may be replaced by the alkyl group which has chloride, bromide, iodide, and cyano \*\* or 1-4 carbon atoms in the location of arbitration);

(iii) The compound of Formula VI [Formula 5]



Or it differs. or [ the inside of a formula, and / that R61, R62, R63, and R64 are the same ] -- It is chosen out of the alkyl group or phenyl group which has 1-10 carbon atoms, respectively, and whether;R65 and R66 are the same Or a difference, It is chosen out of the alkyl group which has a hydrogen atom or 1-10 carbon atoms, respectively. However, as for R65 and R66, when both sides are hydrogen atoms, or both sides are alkyl groups and R65 and R66 are [ both sides ] hydrogen atoms, one or less [ of R61 and R62 ] is a hydrogen atom, and one or less [ of R63 and R64 ] is a hydrogen atom.;

(iv) Compound of Formula VII [Formula 6]



the inside of [type, and R71 -- an oxygen atom or a sulfur atom -- it is -- R75 -- a hydrogen atom or a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) it is chosen out of the alkyl group which has 1-6 carbon atoms, respectively -- it is -- or [ that R72, R73, and R74 are the same ] -- or -- differing -- respectively -- a hydrogen atom -- The alkyl group, phenyl group (replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration) which have 1-6 carbon atoms, And]; chosen from benzyl (the phenyl group may be replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration) and  
(v)-type VIII compound [Formula 7]



since -- the claim chosen -- a solution given in the 1st term.

[Claim 3] (A) A solvent is propylene carbonate.;

A solution contains an inactive current conveyance electrolyte by concentration of 0.01-1.0M in 25 degrees C in a solution. (B) An alkali-metal salt of formula M+31X-35, A formula M+32 Tetra-alkyl ammonium salt of X-35, an aluminum chloride, It is chosen out of combination of aluminium bromide and these salts. M+31 in that case A potassium, sodium or a lithium -- it is -- M+32 -- a tetra-alkylammonium radical (or it differs or [ that an alkyl

group is the same ] --) it is chosen out of an alkyl group which has 1-10 carbon atoms, respectively -- it is -- X-35 -- chloride, bromide, and BF<sub>4</sub><sup>-</sup>, PF<sub>6</sub><sup>-</sup>, AsF<sub>6</sub><sup>-</sup>, ClO<sub>4</sub><sup>-</sup>, or NO<sub>3</sub><sup>-</sup> -- it is --;

(C) A solution contains in a solution one sort of cations with which cathode compounds differ, or two sorts by concentration of 0.01-0.1M in a zero potential balance, respectively by 25 degrees C.;

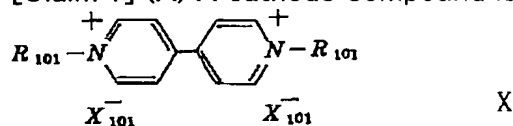
(D) a claim to which a solution contains [ in a solution ] one sort or two sorts of anode compounds by concentration of 0.01-0.1M in a zero potential balance, respectively by 25 degrees C -- a solution given in the 2nd term.

[Claim 4] A solvent is propylene carbonate, cations of a cathode compound are 1 of concentration in [ 0.01-0.1M ] a solution, 1'-dimethyl -4, and 4'-bipyridinium in a zero potential balance at 25 degrees C, an anode compound sets to a zero potential balance at 25 degrees C, and it is 5 of concentration of 0.01-0.1M, and 10-dihydro. - A solution given in the 2nd term of a patent claim which is 5 and 10-dimethyl phenazine.

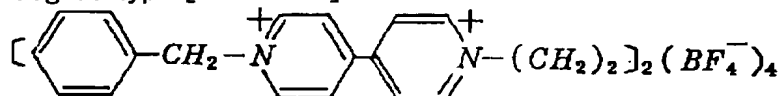
[Claim 5] a claim thickened with 5 - 25% (W/W) of acrylic sheet materials from Lucite (LUCITE) L -- a solution given in the 3rd term.

[Claim 6] a claim thickened with 3 - 20% (W/W) of acrylic sheet materials from Lucite (LUCITE) L -- a solution given in the 4th term.

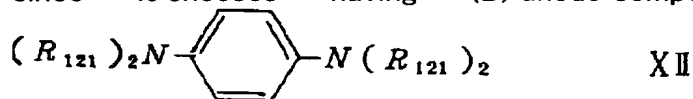
[Claim 7] (A) A cathode compound is a compound of a formula X. [Formula 8]



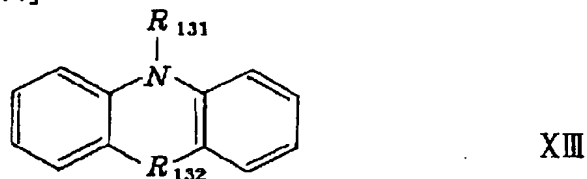
(R101 is n-heptyl, phenyl, or benzyl among a formula, and X-101 is BF<sub>4</sub><sup>-</sup> or ClO<sub>4</sub><sup>-</sup>) And the compound of a degree type [Formula 9]



since -- it chooses -- having -- (B) anode compound -- the compound of Formula XII [Formula 10]



(The inside of a formula and R121 are a methyl group or a phenyl group); and the compound of Formula XIII [Formula 11]



R132 is an oxygen atom, a sulfur atom, or NR133 (R133 is a methyl group or a phenyl group) among {type, and R131 is a methyl group or an ethyl group, when (i) R132 are an oxygen atom or a sulfur atom.;

(ii)); which is a methyl group when R132 is N (CH<sub>3</sub>), and is a phenyl group when R(iii) 132 is N (C<sub>6</sub>H<sub>5</sub>), and compound of Formula VIII [Formula 12]



since -- it chooses -- having -- (C) -- the claim whose inactive current conveyance electrolyte is the compound of a formula M+42 (BF<sub>4</sub><sup>-</sup>) (or [ that the inside M+42 of a formula is a tetra--n-alkylammonium radical, and its n-alkyl group is the same here ] -- or it differs and has 1-6 carbon atoms) -- a solution given in the 3rd term.

[Claim 8] An inactive current conveyance electrolyte is tetra--n-butyl ammonium fluoroborate of concentration of 0.05-0.5M at 25 degrees C. a cathode compound -- 1 and 1' - dibenzyl -4 and 4' -- it is chosen out of - bipyridinium difluoro borate and tetramethylen-screw [4 (1-benzyl pyridine-4'-IRU) pyridinium] tetrafluoroborate -- An anode compound is 5 and 10-dihydro. - 5, 10-dimethyl phenazine and N and N, N', N'-tetramethyl - It is chosen out of 1 and 4-phenylenediamine. A solution given in the 7th term of a patent claim whose total concentration of an anode compound the total concentration of a cathode compound is 0.01-0.05M at 25 degrees C, and is 0.01-0.05M at 25 degrees C.

[Claim 9] As a medium which has reversible adjustable permeability to light, it is the (A) solvent.;

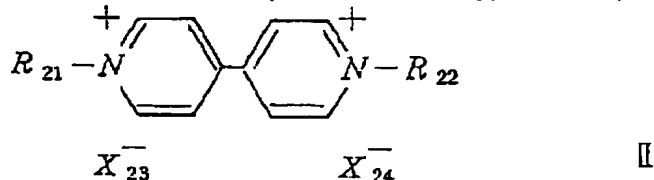
(B) At least one sort of cathode electrochromic compounds accompanied by [ in / among these reduction / in a

voltamogram performed in a room temperature in the above-mentioned solvent, display at least two sorts of chemical reversible reduction waves, and / at least one sort of wavelength of a visible region ] increase of a molar extinction coefficient in the 1st thing;

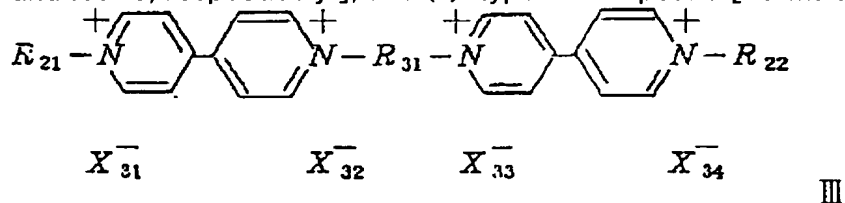
(C) Display at least two sorts of chemical reversible oxidation waves in a voltamogram performed in a room temperature in the above-mentioned solvent. The 1st thing is accompanied by increase of a molar extinction coefficient in at least one sort of wavelength of a visible region among these oxidation. When all of at least one sort of anode electrochromic compound; and (D) cathode compounds, and an anode compound are not ionicity in those zero potential equilibrium in the above-mentioned solvent A single partition mold self-elimination type solution phase electrochromic device which consists of a solution which consists of an inactive current conveyance electrolyte.

[Claim 10] In a solution, the (A) solvent is a liquid at a room temperature, and are two or more sorts of above-mentioned solvents which are liquids at propylene carbonate, gamma-butyrolactone, gamma-valerolactone, and a room temperature, or is chosen from homogeneous mixture of one or more sorts of above-mentioned solvents, and ethylene carbonate.;

(B) A cathode compound is a (i)-type II compound. [Formula 13]

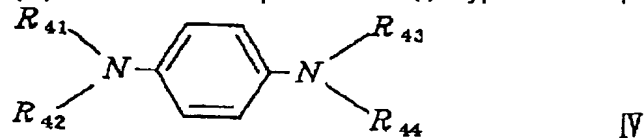


Whether R21 and R22 are the same among [type Or the alkyl group which differ and has 1-10 carbon atoms, respectively, a phenyl group (cyano \*\* the location of arbitration -- setting -- chloride, bromide, and iodide --) Or it is replaced by the alkyl group which has 1-4 carbon atoms, and a potato is good. and benzyl (a phenyl group -- the location of arbitration -- setting -- chloride and bromide --) It is chosen [ that you may be replaced by the alkyl group which has iodide and cyano \*\* or 1-4 carbon atoms and ], and whether;X-23 and X-24 are the same Or a difference, ]; [ which is chosen from chloride, bromide, iodide, BF<sub>4</sub>-, PF<sub>6</sub>-, AsF<sub>6</sub>-, ClO<sub>4</sub>-, and NO<sub>3</sub>-, respectively ], and (ii)-type III compound [Formula 14]



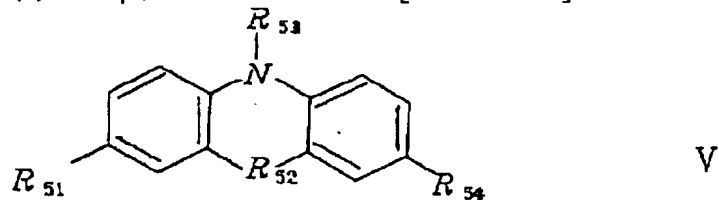
(-- or [ the inside of a formula, and / that R21 and R22 are the same ] -- or -- differing -- previously -- the compound of Formula II -- setting -- R31 -- 1- -- or [ that are the alkylene group which has ten carbon atoms, and X-31, X-32, X-33, and X-34 are the same ] -- or -- differing -- respectively -- chloride, bromide, iodide, and BF<sub>4</sub>- -- it is chosen out of 4, PF<sub>6</sub>-

(C) An anode compound is a (i)-type IV compound. [Formula 15]



Among [type whether R41, R42, R43, and R44 are the same Or a difference, the alkyl group which has a hydrogen atom and 1-10 carbon atoms, respectively, and a phenyl group (the location of arbitration -- setting -- chloride --) You may be replaced by the alkyl group which has bromide, iodide, and cyano \*\* or 1-4 carbon atoms. And] chosen from benzyl (the phenyl group may be replaced by the alkyl group which has chloride, bromide, iodide, and cyano \*\* or 1-4 carbon atoms in the location of arbitration);

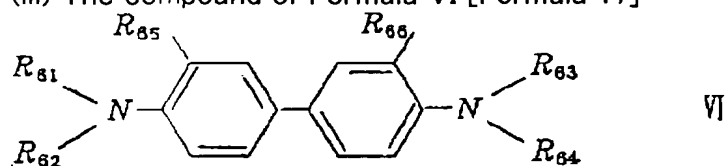
(ii) Compound of Formula V [Formula 16]



or [ the inside of [type, and / that R51 and R54 are the same ] -- or -- differing -- respectively -- a hydrogen atom and a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) It is chosen out of having

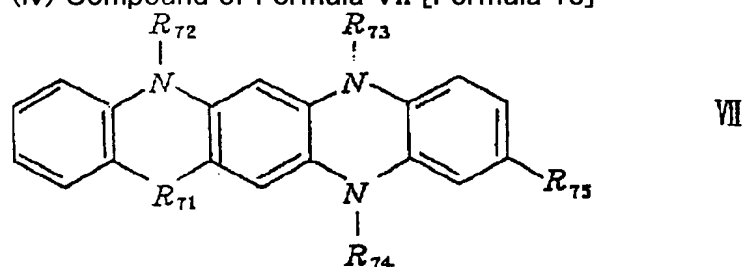
1-6 carbon atoms, respectively.;R52 An oxygen atom, Whether it is a sulfur atom or NR55, and R55 is the same as that of R53 here Or a difference, The alkyl group in which both R55 and R53 have a hydrogen atom and 1-10 carbon atoms, a phenyl group (cyano \*\* the location of arbitration -- setting -- chloride, bromide, and iodide --) Or you may be replaced by the alkyl group which has 1-4 carbon atoms. Or} chosen from benzyl (the phenyl group may be replaced by the alkyl group which has chloride, bromide, iodide, and cyano \*\* or 1-4 carbon atoms in the location of arbitration);

(iii) The compound of Formula VI [Formula 17]



Or it differs. or [ the inside of a formula, and / that R61, R62, R63, and R64 are the same ] -- It is chosen out of the alkyl group or phenyl group which has 1-10 carbon atoms, respectively, and whether;R65 and R66 are the same Or a difference, It is chosen out of the alkyl group which has a hydrogen atom or 1-10 carbon atoms, respectively. However, as for R65 and R66, when both sides are hydrogen atoms, or both sides are alkyl groups and R65 and R66 are [ both sides ] hydrogen atoms, one or less [ of R61 and R62 ] is a hydrogen atom, and one or less [ of R63 and R64 ] is a hydrogen atom.;

(iv) Compound of Formula VII [Formula 18]



the inside of {type, and R71 -- an oxygen atom or a sulfur atom -- it is -- R75 -- a hydrogen atom or a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) it is chosen out of the alkyl group which has 1-6 carbon atoms, respectively -- it is -- or [ that R72, R73, and R74 are the same ] -- or -- differing -- respectively -- a hydrogen atom -- The alkyl group, phenyl group (replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration) which have 1-6 carbon atoms, And}; chosen from benzyl (the phenyl group may be replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration) and (v)-type VIII compound [Formula 19]



since -- the claim chosen -- a device given in the 9th term.

[Claim 11] In a solution, the (A) solvent is propylene carbonate.;

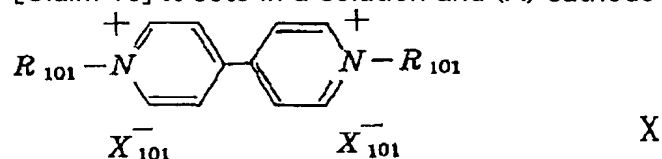
A solution contains an inactive current conveyance electrolyte by concentration of 0.01-1.0M in 25 degrees C in a solution. (B) An alkali-metal salt of formula M+31X-35, A formula M+32 Tetra-alkyl ammonium salt of X-35, an aluminum chloride, It is chosen out of combination of aluminium bromide and these salts. M+31 in that case A potassium, sodium or a lithium -- it is -- M+32 -- a tetra-alkylammonium radical (or it differs or [ that an alkyl group is the same ] --) it is chosen out of an alkyl group which has 1-10 carbon atoms, respectively -- it is -- X-35 -- chloride, bromide, and BF- 4, PF-6, AsF-6, ClO-4, or NO-3 -- it is --;

(C) Contain [ in one sort of cations with which cathode compounds differ, or two sorts of solutions ] a solution by concentration of 0.01-0.1M in a zero potential balance, respectively by 25 degrees C.;

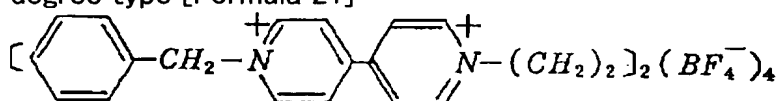
(D) a claim to which a solution contains [ in a solution ] one sort or two sorts of anode compounds by concentration of 0.01-0.1M in a zero potential balance, respectively by 25 degrees C -- a device given in the 10th term.

[Claim 12] a claim by which a solution was thickened with 5 - 25% (w/w) of acrylic sheet materials from Lucite (LUCITE) L -- a device given in the 11th term.

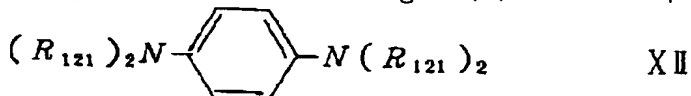
[Claim 13] It sets in a solution and (A) cathode compound is a compound of a formula X. [Formula 20]



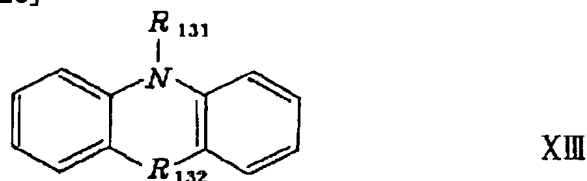
(R101 is n-heptyl, phenyl, or benzyl among a formula, and X-101 is BF<sub>4</sub><sup>-</sup> or ClO<sub>4</sub><sup>-</sup>) And the compound of a degree type [Formula 21]



since -- it chooses -- having -- (B) anode compound -- the compound of Formula XII [Formula 22]



(The inside of a formula and R121 are a methyl group or a phenyl group); and the compound of Formula XIII [Formula 23]



R132 is an oxygen atom, a sulfur atom, or NR133 (R133 is a methyl group or a phenyl group) among {type, and R131 is a methyl group or an ethyl group, when (i) R132 are an oxygen atom or a sulfur atom.;

(ii)); which is a methyl group when R132 is N (CH3), and is a phenyl group when R(iii) 132 is N (C6H5), and compound of Formula VIII [Formula 24]



since -- it chooses -- having -- (C) -- the claim whose inactive current conveyance electrolyte is the compound of a formula M+42 (BF<sub>4</sub><sup>-</sup>) (or [ that the inside M+42 of a formula is a tetra--n-alkylammonium radical, and its n-alkyl group is the same here ] -- or it differs and has 1-6 carbon atoms) -- a device given in the 11th term.

[Claim 14] In a solution, an inactive current conveyance electrolyte is tetra--n-butyl ammonium fluoroborate of concentration of 0.05-0.5M at 25 degrees C. A cathode compound is chosen from 1, 1'-dibenzyl -4, 4'-bipyridinium difluoro borate, and tetramethylen-screw [4-(1-benzyl pyridine-4'-IRU) pyridinium] tetrafluoroborate. An anode compound is 5 and 10-dihydro. - 5, 10-dimethyl phenazine and N and N, N', N'-tetramethyl - It is chosen out of 1 and 4-phenylenediamine. A device given in the 11th term of a patent claim whose total concentration of an anode compound the total concentration of a cathode compound is 0.01-0.05M at 25 degrees C, and is 0.01-0.05M at 25 degrees C.

[Claim 15] As a medium which has reversible adjustable permeability to light reflected by mirror, it is the (A) solvent.;

(B) At least one sort of cathode electrochromic compounds accompanied by [ in / among these reduction / in a voltamogram performed in a room temperature in the above-mentioned solvent, display at least two sorts of chemical reversible reduction waves, and / at least one sort of wavelength of a visible region ] increase of a molar extinction coefficient in the 1st thing;

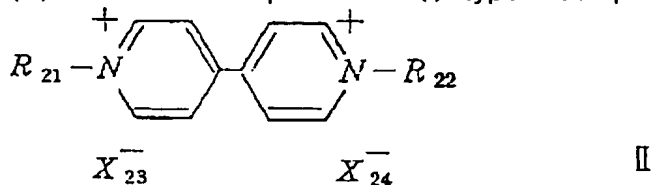
(C) Display at least two sorts of chemical reversible oxidation waves in a voltamogram performed in a room temperature in the above-mentioned solvent. The 1st thing is accompanied by increase of a molar extinction coefficient in at least one sort of wavelength of a visible region among these oxidation. When all of at least one sort of anode electrochromic compound; and (D) cathode compounds, and an anode compound are not ionicity in those zero potential equilibrium in the above-mentioned solvent An adjustable permeability mirror which consists of a single partition mold self-elimination type solution phase electrochromic device which consists of a solution which consists of an inactive current conveyance electrolyte.

[Claim 16] In an electrochromic device, a solution with reversible adjustable permeability by flatness of two sheets [ parallel ] It is placed by the condition of having contacted these, between walls which kept a gap, among those at least one side is transparent. A side to which they contacted the above-mentioned solution, respectively is covered with an electrode layer of a conductive material. However, it is a mirror given in the 15th term of a patent claim which reflects light which reaches a reflecting layer after this advances into a solution through a transparent wall through a solution and a transparent wall by this consisting of a reflecting layer of a high reflection factor material among those walls when one side is not transparent.

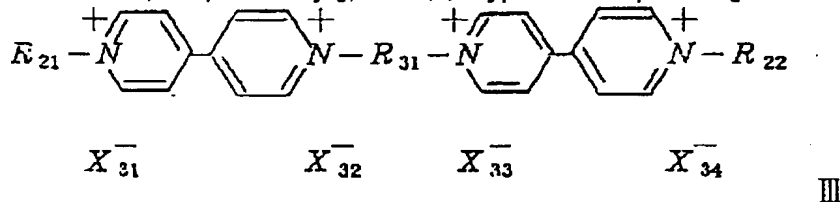
[Claim 17] In a solution with reversible adjustable permeability, the (A) solvent is a liquid at a room temperature,

and are two or more sorts of above-mentioned solvents which are liquids at propylene carbonate, gamma-butyrolactone, gamma-valerolactone, and a room temperature, or is chosen from homogeneous mixture of one or more sorts of above-mentioned solvents, and ethylene carbonate.;

(B) A cathode compound is a (i)-type II compound. [Formula 25]

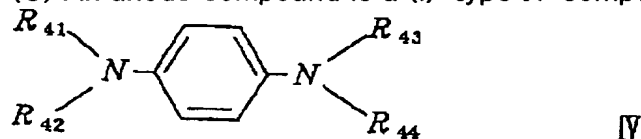


Whether R21 and R22 are the same among [type Or the alkyl group which differ and has 1-10 carbon atoms, respectively, a phenyl group (cyano \*\* the location of arbitration -- setting -- chloride, bromide, and iodide --) Or it is replaced by the alkyl group which has 1-4 carbon atoms, and a potato is good. and benzyl (a phenyl group -- the location of arbitration -- setting -- chloride and bromide --) It is chosen [ that you may be replaced by the alkyl group which has iodide and cyano \*\* or 1-4 carbon atoms and ], and whether;X-23 and X-24 are the same Or a difference, ]; [ which is chosen from chloride, bromide, iodide, BF-4, PF-6, AsF-6, ClO-4, and NO-3, respectively ], and (ii)-type III compound [Formula 26]



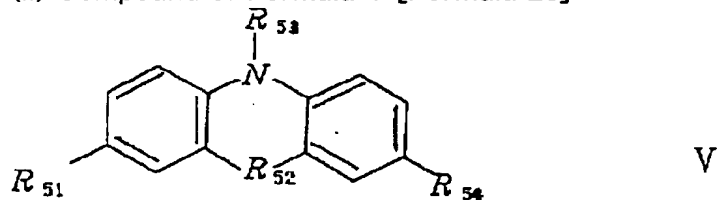
(-- or [ the inside of a formula, and / that R21 and R22 are the same ] -- or -- differing -- previously -- the compound of Formula II -- setting -- R31 -- 1- -- or [ that are the alkylene group which has ten carbon atoms, and X-31, X-32, X-33, and X-34 are the same ] -- or -- differing -- respectively -- chloride, bromide, iodide, and BF- -- it is chosen out of 4, PF-6

(C) An anode compound is a (i)-type IV compound. [Formula 27]



Among [type whether R41, R42, R43, and R44 are the same Or a difference, the alkyl group which has a hydrogen atom and 1-10 carbon atoms, respectively, and a phenyl group (the location of arbitration -- setting -- chloride --) You may be replaced by the alkyl group which has bromide, iodide, and cyano \*\* or 1-4 carbon atoms. And} chosen from benzyl (the phenyl group may be replaced by the alkyl group which has chloride, bromide, iodide, and cyano \*\* or 1-4 carbon atoms in the location of arbitration);

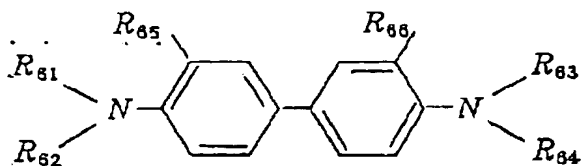
(ii) Compound of Formula V [Formula 28]



or [ the inside of [type, and / that R51 and R54 are the same ] -- or -- differing -- respectively -- a hydrogen atom and a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) It is chosen out of having 1-6 carbon atoms, respectively.;R52 An oxygen atom, Whether it is a sulfur atom or NR55, and R55 is the same as that of R53 here Or a difference, The alkyl group in which both R55 and R53 have a hydrogen atom and 1-10 carbon atoms, a phenyl group (cyano \*\* the location of arbitration -- setting -- chloride, bromide, and iodide --) Or you may be replaced by the alkyl group which has 1-4 carbon atoms. Or} chosen from benzyl (the phenyl group may be replaced by the alkyl group which has chloride, bromide, iodide, and cyano \*\* or 1-4 carbon atoms in the location of arbitration);

(iii) The compound of Formula VI [Formula 29]

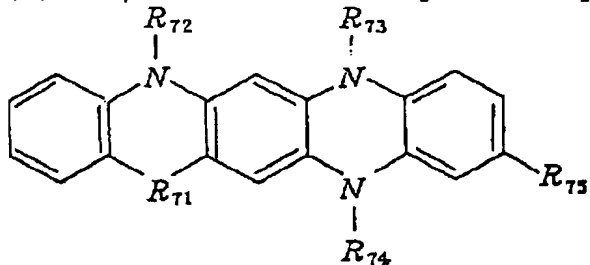




VI

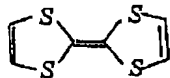
Or it differs. or [ the inside of a formula, and / that R61, R62, R63, and R64 are the same ] -- It is chosen out of the alkyl group or phenyl group which has 1-10 carbon atoms, respectively, and whether; R65 and R66 are the same Or a difference, It is chosen out of the alkyl group which has a hydrogen atom or 1-10 carbon atoms, respectively. However, as for R65 and R66, when both sides are hydrogen atoms, or both sides are alkyl groups and R65 and R66 are [ both sides ] hydrogen atoms, one or less [ of R61 and R62 ] is a hydrogen atom, and one or less [ of R63 and R64 ] is a hydrogen atom.;

(iv) Compound of Formula VII [Formula 30]



VII

the inside of [type, and R71 -- an oxygen atom or a sulfur atom -- it is -- R75 -- a hydrogen atom or a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) it is chosen out of the alkyl group which has 1-6 carbon atoms, respectively -- it is -- or [ that R72, R73, and R74 are the same ] -- or -- differing -- respectively -- a hydrogen atom -- The alkyl group, phenyl group (replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration) which have 1-6 carbon atoms, And]; chosen from benzyl (the phenyl group may be replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration) and (v)-type VIII compound [Formula 31]



VIII

since -- the claim chosen -- a mirror given in the 16th term.

[Claim 18] In a solution with reversible adjustable permeability, the (A) solvent is propylene carbonate.;

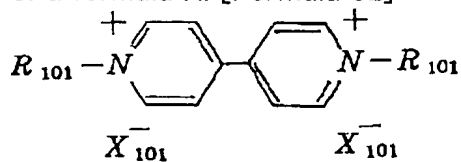
A solution contains an inactive current conveyance electrolyte by concentration of 0.01-1.0M in 25 degrees C in a solution. (B) An alkali-metal salt of formula M+31X-35, A formula M+32 Tetra-alkyl ammonium salt of X-35, an aluminum chloride, It is chosen out of combination of aluminium bromide and these salts. M+31 in that case A potassium, sodium or a lithium -- it is -- M+32 -- a tetra-alkylammonium radical (or it differs or [ that an alkyl group is the same ] --) it is chosen out of an alkyl group which has 1-10 carbon atoms, respectively -- it is -- X-35 -- chloride, bromide, and BF- 4, PF-6, AsF-6, ClO-4, or NO-3 -- it is --;

(C) Contain [ in one sort of cations with which cathode compounds differ, or two sorts of solutions ] a solution by concentration of 0.01-0.1M in a zero potential balance, respectively by 25 degrees C.;

(D) a claim to which a solution contains [ in a solution ] one sort or two sorts of anode compounds by concentration of 0.01-0.1M in a zero potential balance, respectively by 25 degrees C -- a mirror given in the 17th term.

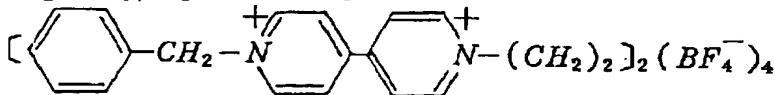
[Claim 19] a claim by which a solution with reversible adjustable permeability was thickened with 5 - 25% (w/w) of acrylic sheet materials from Lucite (LUCITE) L -- a mirror given in the 18th term.

[Claim 20] It sets in a solution with reversible adjustable permeability, and (A) cathode compound is a compound of a formula X. [Formula 32]

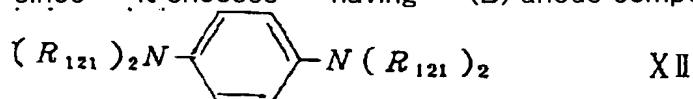


X

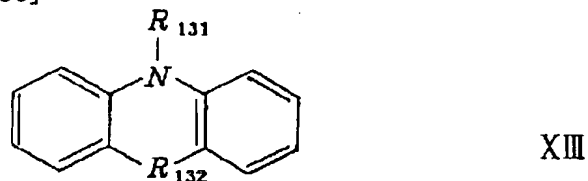
(R101 is n-heptyl, phenyl, or benzyl among a formula, and X-101 is BF-4 or ClO-4) And the compound of a degree type [Formula 33]



since -- it chooses -- having -- (B) anode compound -- the compound of Formula XII [Formula 34]



(The inside of a formula and R121 are a methyl group or a phenyl group); and the compound of Formula XIII [Formula 35]



R132 is an oxygen atom, a sulfur atom, or NR133 (R133 is a methyl group or a phenyl group) among {type, and R131 is a methyl group or an ethyl group, when (i) R132 are an oxygen atom or a sulfur atom; (ii)}; which is a methyl group when R132 is N (CH3), and is a phenyl group when R(iii) 132 is N (C6H5), and compound of Formula VIII [Formula 36]



since -- it chooses -- having -- (C) -- the claim whose inactive current conveyance electrolyte is the compound of a formula M+42 (BF-4) (or [ that the inside M+42 of a formula is a tetra--n-alkylammonium radical, and its n-alkyl group is the same here ] -- or it differs and has 1-6 carbon atoms) -- a mirror given in the 18th term.

[Claim 21] In a solution with reversible adjustable permeability, an inactive current conveyance electrolyte is tetra--n-butyl ammonium fluoroborate of concentration of 0.05-0.5M at 25 degrees C. A cathode compound is chosen from 1, 1'-dibenzyl -4, 4'-bipyridinium difluoro borate, and tetramethylen-screw [4-(1-benzyl pyridine-4'-IRU) pyridinium] tetrafluoroborate. An anode compound is 5 and 10-dihydro. - 5, 10-dimethyl phenazine and N and N, N', N'-tetramethyl - It is chosen out of 1 and 4-phenylenediamine. A mirror given in the 20th term of a patent claim whose total concentration of an anode compound the total concentration of a cathode compound is 0.01-0.05M at 25 degrees C, and is 0.01-0.05M at 25 degrees C.

[Claim 22] (A) It is glass equipped with an electrode layer of tin oxide in contact with a solution in which an electrode possession wall of both sides of an electrochromic device has reversible adjustable permeability which carried out the indium dope. A gap of an electrode layer is 0.006-0.025cm, and; and (B), and (i) one wall is transparent to light of visible wavelength. These walls are [ as opposed to / in both sides / light of visible wavelength ] transparent. or, as for a wall of another side, an electrode possession side and a field of the opposite side are covered with a reflecting layer --; or (ii) -- A reflective means of a mirror is the high reflector of a prism form mirror in which the laminating was carried out to one side of these walls by transparent charge of a laminated wood. ; An angle of a high reflector of a prism form mirror and a laminating side is 1-5 degrees, and correct it. When one electrode possession wall is covered with a reflecting layer, the laminating of the electrode possession wall of another side may be carried out to a glass piece of a prism form by transparent charge of a laminated wood. It is a written mirror to the 16th term of a patent claim whose angle of a field by which the laminating was carried out to an electrode possession wall at that time, and a field of another side through which light which reached an electrode possession wall passes altogether substantially is 1-5 degrees. [Claim 23] A mirror given in the 22nd term of a patent claim which is the high reflector of a prism form mirror where the laminating of the reflective means was carried out to one side of an electrode possession wall of an electrochromic device.

[Claim 24] (A) It is glass equipped with an electrode layer of tin oxide in contact with a solution in which an electrode possession wall of both sides of an electrochromic device has reversible adjustable permeability which carried out the indium dope. A gap of an electrode layer is 0.006-0.025cm, and; and (B), and (i) one wall is transparent to light of visible wavelength. These walls are [ as opposed to / in both sides / light of visible wavelength ] transparent. or, as for a wall of another side, an electrode possession side and a field of the opposite side are covered with a reflecting layer --; or (ii) -- A reflective means of a mirror is the high reflector of a prism form mirror in which the laminating was carried out to one side of these walls by transparent charge of a laminated wood. ; An angle of a high reflector of a prism form mirror and a laminating side is 1-5 degrees, and correct it. When one electrode possession wall is covered with a reflecting layer, the laminating of the electrode possession wall of another side may be carried out to a glass piece of a prism form by transparent charge of a laminated wood. A mirror given in the 17th term of a patent claim whose angle of a field by which the laminating was carried out to an electrode possession wall at that time, and a field of another side through which

light which reached an electrode possession wall passes altogether substantially is 1–5 degrees.

[Claim 25] A mirror given in the 24th term of a patent claim which is the high reflector of a prism form mirror where the laminating of the reflective means was carried out to one side of an electrode possession wall of an electrochromic device.

[Claim 26] (A) It is glass equipped with an electrode layer of tin oxide in contact with a solution in which an electrode possession wall of both sides of an electrochromic device has reversible adjustable permeability which carried out the indium dope. A gap of an electrode layer is 0.006–0.025cm, and; and (B), and (i) one wall is transparent to light of visible wavelength. These walls are [ as opposed to / in both sides / light of visible wavelength ] transparent. or, as for a wall of another side, an electrode possession side and a field of the opposite side are covered with a reflecting layer --; or (ii) -- A reflective means of a mirror is the high reflector of a prism form mirror in which the laminating was carried out to one side of these walls by transparent charge of a laminated wood. ; An angle of a high reflector of a prism form mirror and a laminating side is 1–5 degrees, and correct it. When one electrode possession wall is covered with a reflecting layer, the laminating of the electrode possession wall of another side may be carried out to a glass piece of a prism form by transparent charge of a laminated wood. A mirror given in the 18th term of a patent claim whose angle of a field by which the laminating was carried out to an electrode possession wall at that time, and a field of another side through which light which reached an electrode possession wall passes altogether substantially is 1–5 degrees.

[Claim 27] A mirror given in the 26th term of a patent claim which is the high reflector of a prism form mirror where the laminating of the reflective means was carried out to one side of an electrode possession wall of an electrochromic device.

[Claim 28] (A) It is glass equipped with an electrode layer of tin oxide in contact with a solution in which an electrode possession wall of both sides of an electrochromic device has reversible adjustable permeability which carried out the indium dope. A gap of an electrode layer is 0.006–0.025cm, and; and (B), and (i) one wall is transparent to light of visible wavelength. These walls are [ as opposed to / in both sides / light of visible wavelength ] transparent. or, as for a wall of another side, an electrode possession side and a field of the opposite side are covered with a reflecting layer --; or (ii) -- A reflective means of a mirror is the high reflector of a prism form mirror in which the laminating was carried out to one side of these walls by transparent charge of a laminated wood. ; An angle of a high reflector of a prism form mirror and a laminating side is 1–5 degrees, and correct it. When one electrode possession wall is covered with a reflecting layer, the laminating of the electrode possession wall of another side may be carried out to a glass piece of a prism form by transparent charge of a laminated wood. A mirror given in the 19th term of a patent claim whose angle of a field by which the laminating was carried out to an electrode possession wall at that time, and a field of another side through which light which reached an electrode possession wall passes altogether substantially is 1–5 degrees.

[Claim 29] A mirror given in the 28th term of a patent claim which is the high reflector of a prism form mirror where the laminating of the reflective means was carried out to one side of an electrode possession wall of an electrochromic device.

[Claim 30] (A) It is glass equipped with an electrode layer of tin oxide in contact with a solution in which an electrode possession wall of both sides of an electrochromic device has reversible adjustable permeability which carried out the indium dope. A gap of an electrode layer is 0.006–0.025cm, and; and (B), and (i) one wall is transparent to light of visible wavelength. These walls are [ as opposed to / in both sides / light of visible wavelength ] transparent. or, as for a wall of another side, an electrode possession side and a field of the opposite side are covered with a reflecting layer --; or (ii) -- A reflective means of a mirror is the high reflector of a prism form mirror in which the laminating was carried out to one side of these walls by transparent charge of a laminated wood. ; An angle of a high reflector of a prism form mirror and a laminating side is 1–5 degrees, and correct it. When one electrode possession wall is covered with a reflecting layer, the laminating of the electrode possession wall of another side may be carried out to a glass piece of a prism form by transparent charge of a laminated wood. A mirror given in the 20th term of a patent claim whose angle of a field by which the laminating was carried out to an electrode possession wall at that time, and a field of another side through which light which reached an electrode possession wall passes altogether substantially is 1–5 degrees.

[Claim 31] A mirror given in the 30th term of a patent claim which is the high reflector of a prism form mirror where the laminating of the reflective means was carried out to one side of an electrode possession wall of an electrochromic device.

[Claim 32] (A) It is glass equipped with an electrode layer of tin oxide in contact with a solution in which an electrode possession wall of both sides of an electrochromic device has reversible adjustable permeability which carried out the indium dope. A gap of an electrode layer is 0.006–0.025cm, and; and (B), and (i) one wall is transparent to light of visible wavelength. These walls are [ as opposed to / in both sides / light of visible wavelength ] transparent. or, as for a wall of another side, an electrode possession side and a field of the opposite side are covered with a reflecting layer --; or (ii) -- A reflective means of a mirror is the high reflector of a prism form mirror in which the laminating was carried out to one side of these walls by transparent charge

of a laminated wood. ; An angle of a high reflector of a prism form mirror and a laminating side is 1–5 degrees, and correct it. When one electrode possession wall is covered with a reflecting layer, the laminating of the electrode possession wall of another side may be carried out to a glass piece of a prism form by transparent charge of a laminated wood. A mirror given in the 21st term of a patent claim whose angle of a field by which the laminating was carried out to an electrode possession wall at that time, and a field of another side through which light which reached an electrode possession wall passes altogether substantially is 1–5 degrees.  
[Claim 33] A mirror given in the 32nd term of a patent claim which is the high reflector of a prism form mirror where the laminating of the reflective means was carried out to one side of an electrode possession wall of an electrochromic device.

---

[Translation done.]

## \* NOTICES \*

Japan Patent Office is not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

## DETAILED DESCRIPTION

---

### [Detailed Description of the Invention]

[0001] this invention -- electromagnetism -- it is related with using the device which has reversible adjustable permeability to a line, the constituents used as a medium which has reversible adjustable permeability in this kind of device, and these devices as an adjustable permeability color filter and an adjustable reflection factor mirror. This invention relates to a single partition mold self-elimination type solution electrochromic device and the solution used for these more at details.

[0002] electromagnetism -- several sorts of devices into which the permeability to a line is reversibly changeable are known. the electromagnetism which permeability is changed into this kind of device with thermochromic, photochromic one, or an electro-optics--(for example, liquid crystal, bipolar suspension (dipolar suspension), electromotive type (electrophoretic), electrochromic) means, and has adjustable permeability in a visible region partially at least -- the device which is a thing to a line (wavelength of 4200-7000A) is contained.

[0003] electromagnetism -- the device which has reversible adjustable permeability to a line is used as an adjustable permeability element in the display device which uses an adjustable permeability color filter, an adjustable reflection factor mirror, and this kind of a color filter or a mirror for informational conveyance. An aperture is contained in these adjustable permeability color filters. The glare prevention mold reflector glass for automobiles is contained in an adjustable reflection factor mirror.

[0004] the electromagnetism into which permeability is changed by the electrochromic means -- an erection ROKEMIKUROMIKKU device is contained in a device with the reversible adjustable permeability to a line, and this is indicated by "electrochromic one, electrochromic materials, and the phenomenon" (the volume a non-radiating mold electro-optics display, A KUMETTSU, and for KE Von Willy Seng, a par gamone press, New York State New York, 1976, 155-196 pages, 1976) by Chillan. That in which electrochemical reaction occurs in a solid-state film, the thing accompanied by electroplating, or the thing which happens in a solution on the whole is contained in an electrochromic device. Please refer to the reference of above-mentioned Chillan.

[0005] Many electrochromic devices are known for this technical field. for example, MANOSU et al., U.S. Pat. No. 3,451,741; bread felt, U.S. Pat. No. 4,090,782; SHIYA@KKU and SHINSA box, and U.S. Pat. No. 4,093,358; -- KUREKAKU et al. and U.S. Pat. No. 4,139,276; -- KISA et al., U.S. Pat. No. 3,453,038; low jars, U.S. Pat. No. 3,652,149, No. 3,774,988, and No. 3,873,185; -- please refer to Jones et al., U.S. Pat. No. 3,282,157, No. 3,282,158, No. 3,282,160, and No. 3,283,656 each specification. A single mold self-elimination type solution phase electrochromic device is contained in these devices. above-mentioned MANOSU(it quotes as reference here); -- bread felt; -- please refer to the specification of SHIYA@KKU and SHINSA box, and KUREKAKU and others.

[0006] passing the solution held in the partition containing two electrodes of a device in the single partition mold self-elimination type solution phase electrochromic device -- electromagnetism -- the reinforcement of a line is modulated. These 2 electrode touches the solution. The partition of a solution is divided into inter-electrode and there is no barrier which prevents that a certain component in a solution spreads or moves to another side from one electrode, for example, semipermeable membrane etc., in it. A solution contains a solvent and at least one sort of compound (also neutrally, charged) and at least 1 sort of "anode" "cathode" compounds (also neutrally, this may also be charged). If DC potential difference is given to an inter-electrode solution, an "anode" compound will oxidize electrochemically and a "cathode" compound will be returned electrochemically. Probably, the solution contains the inactive current conveyance electrolyte, when the "anode" compound and "cathode" compound oxidized or returned all is not charged before oxidation or reduction, respectively. Or you may contain. As for each of other components considered to exist in a solvent, an inactive current conveyance electrolyte (when contained), an anode compound, a cathode compound, and a solution, it is desirable that it is that to which it sets to the inter-electrode potential difference which does not cause other electrochemical change or other change significant for a component in a solution, and an anode compound and a cathode compound are oxidized and returned, respectively. The solution is liquefied during actuation of a device. However, this is gelled, or is made hyperviscosity by addition of a thickener, and a potato is good. That a device is a "solution phase" means that all the components in a solution including an anode compound and a cathode compound are maintained in the shape of a solution during actuation of the device accompanied by oxidation of an anode compound, and

reduction of a cathode compound.

[0007] the electromagnetism which passes a single partition mold self-elimination type solution phase electrochromic device -- the reversible modulation of the reinforcement of a line can be attained for three factors relevant to actuation of a device. The molar extinction coefficient (that of wavelength is a function) of the anode compound in the solution of a device and a cathode compound changes [ 1st ] with those electrochemical oxidation and reduction, respectively. Generally an absorbancy index changes [ in / on the occasion of oxidation or reduction / in at least one sort / the wavelength of a visible region ] intentionally among these compounds. Consequently, if the potential difference is given to an inter-electrode solution, the color of a solution and a device will change or it will change from \*\* from dark, or \*\* to dark. The anode compound and the returned cathode compound which oxidized in the solution to the 2nd cause a decomposition reaction for neither of other components as single molecules to a significant degree. The anode compound which oxidized in the solution to the 3rd reacts substantially only with the returned cathode compound, and only the anode compound and cathode compound which were equipped with those gestalten and those properties before oxidation and reduction, respectively are given substantially. "Self-elimination" (selferasing)" nature is given to a device by this reaction of the oxidized anode compound and the returned cathode compound.

[0008] The single partition mold self-elimination type solution phase electrochromic device with which it was proved that it is suitable to use it commercially until now as a component part which has reversible adjustable permeability in an adjustable permeability color filter or an adjustable reflective mirror was not known. For this kind of use, thousands of times at least must be stable to the circulation which the solution with adjustable permeability changes from an inter-electrode zero potential difference to the inter-electrode potential difference of sufficient degree to change permeability intentionally, and returns again subsequently to zero. in a typical device, a solution is held in the shape of a layer between the transparent walls which kept the flat and parallel gap, and the thin layer of a transparent conductive material applies it to the inside (contacting a solution) of a wall -- having -- \*\*\*\* -- these -- as an electrode -- acting -- this -- electromagnetism -- a line passes and that reinforcement is reversibly modulated within this device. In order to shorten the "response time" required in order to suppress the strain of light which passes a device and is reflected from a device to the minimum, and in order [ when the inter-electrode potential difference changes, ] to attain permeability with the new permeability of a device even to time amount permissible in commercial use, it is advantageous to make a solution layer as thin as possible. however, for a device with a dilute-solution layer In the concentration in the solution maintained at fusibility on the both sides in the condition of having been oxidized and (in the case of an anode compound) returned when the potential difference was given to zero potential equilibrium and inter-electrode (in the case of a cathode compound) Change of the absorbance of degree sufficient between "(that is, oxidized or returned) conditions of which "activation was done with zero potential equilibrium is produced. The anode and cathode electrochromic compound which maintain a stable condition to circulation must be found out, so that it is enough to give the device which has practicality in coincidence commercially. This invention tackles the request to the solution for creating the single partition mold self-elimination type solution phase electrochromic device which is commercially practical.

[0009] The useful special feature which was not acquired in this kind of device until now is that the permeability of the light of a visible wavelength field may change continuously and promptly as a function of functioning as a gray-scale device, i.e., the potential difference given to inter-electrode [ of a device ]. This kind of "gray-scale" device could be used for the glare prevention reflector glass for automobiles which reflects the light of reinforcement permissible to an operator in the aperture which passes the light of fixed reinforcement regardless of the luminous intensity which reaches an aperture regardless of the luminous intensity which carries out incidence to a mirror from the headlight of the automobile which approaches an automobile from back again, and which produces a glare. This invention offers the gray-scale ability in a single partition mold self-elimination type solution phase electrochromic device.

[0010] The problems which have not been recognized about the solution phase electrochromic device until now are migration of an anode and a cathode electrochromic compound and the segregation by the free convection. Where the plane where light goes into a device through it especially is perpendicularly arranged to the ground, in the device by which long duration (20 minutes or more) consecutive operation is carried out, the fall of troublesome color separation and self-erasing speed may take place by such segregation. This invention tackles the problem of this segregation.

[0011] A component part (it is the device from which the permeability to a visible ray changes with thermochromic, photochromic one, or electron optics-means reversibly) with adjustable permeability, and a reflective means (after passing a medium with the reversible adjustable permeability of a component part with adjustable permeability, light is the high reflector reflected from now on, for example, a silver larer) are included in an adjustable reflection factor mirror. After being reflected from a reflective means, this reflected light carries out execution passage of the medium with reversible adjustable permeability. Generally the adjustable permeability medium in this kind of mirror is held in an adjustable permeability component part between the

surfaces in which the parallel gap was kept by the flatness of two sheets. At least one side is transparent to light among these surfaces, and the light reflected by the mirror advances into this transparent surface, and it passes through it. The problem about this kind of mirror is high "residual" reflective power, and this is 5% or more of the transparent surface of the component part which usually has adjustable permeability. For example, in order to remove a high glare, when it is the glare prevention reflector glass for automobiles considered that it is necessary to decrease the reflective power from all the surfaces that an operator looks at to about 5 - 7%, since the residual reflective power of the front face of a typical mirror is high, it is necessary to reduce the permeability of the reversible adjustable permeability medium in a mirror to about 3%, and to deal in it. Thus, probably, as for attaining low permeability at sufficient speed in a thin device to a desirable degree with reversible adjustable permeability, it will be advantageous to obtain the adjustable reflection factor mirror by which these problems produced with high residual reflective power are avoided, since it is difficult. This invention offers this kind of mirror.

[0012] this invention -- a single partition mold self-elimination type solution phase electrochromic device -- setting -- electromagnetism -- the solution used as a medium which has reversible adjustable permeability to a line (especially light of a visible region) is offered.

[0013] The electrochromic device with which, as for this invention, the solution of this invention is further used as a reversible adjustable permeability medium; the display device with which adjustable permeability color filter and adjustable reflection factor mirror; and information that a component part with adjustable permeability is a single mold self-elimination type solution phase device by this invention are displayed by actuation of the adjustable permeability color filter by this invention or an adjustable reflection factor mirror is offered.

[0014] The solution of this invention enables commercially implementation of use of the display device which uses a single partition mold self-elimination type solution phase electrochromic device and an adjustable permeability color filter, an adjustable reflection factor mirror, and this kind of a filter and a mirror. The solution of this invention shows high stability unexpectedly to circulation of the inter-electrode potential difference in the device of this invention.

[0015] A solution layer is thin to a desirable degree, and are so low that the problem of segregation is enough to decrease substantially, without the concentration of the anode compound in a solution and a cathode compound producing precipitate. Moreover, it sets to the device of low this invention a forge fire enough that the inter-electrode potential difference avoids significant disassembly of a solution. If it will dark-color-ize even to an unexpectedly high absorbance to a visible ray at high speed unexpectedly if the potential difference is given, and it open-circuit-forms and an electrode short-circuits, light coloring of the solution of this invention will be carried out again unexpectedly at high speed. In order to cause light coloring at speed sufficient for many practical uses, it is not necessary to make an advantageous thing reverse the polarity of the electrode of the device of this invention. Furthermore, the device of this invention can operate advantageously as a gray-scale device.

[0016] In other viewpoints, this invention is accompanied by the combination of a new electrochromic compound and a compound, in order to use it for the solution of this invention.

[0017] the improved adjustable reflection factor mirror contains in this invention in the viewpoint of further others -- having -- that case -- an adjustable reflection factor -- electromagnetism -- in a device with the adjustable permeability to a line, it is given by thermochromic, photochromic one, or the electro-optics-means. In the mirror of this invention by which this kind was improved, the problem which originates in residual reflective power from the plane out of which it comes from a mirror after light advances through it and reflects from a reflective means is avoided by [ slight ] shifting the degree of angle to the high plane of reflection which is the reflective means of a mirror in this plane. It becomes unnecessary thereby, for those who look at a mirror to look at the light by residual reflective power to the light and coincidence which were reflected from the reflective means of a mirror.

[0018] A drawing is explained briefly.

[0019] the condition that drawing 1 left the electrode possession side plate in the transparent electrode possession side plates 100 and 130 and the assembled device of this invention by the flatness of two sheets of the device of this invention -- and the exploded view of the spacer held in parallel substantially or the separation means 11 is shown roughly. The common-law marriages 11A, 11B, 11C, and 11D of a spacer appoint space 12 with the electrode layers 10A and 13A of an electrode possession side plate. This is occupied by the solution of this invention in contact with an electrode layer in the assembled device of this invention.

[0020] Drawing 2 shows roughly the part assembly drawing of the device 200 by this invention. Drawing 2 shows side plate 100 transparent portion by the flatness of a device by the field 14 which gave the slash. A color changes in connection with a cover, therefore a device operating the solution which has adjustable reversible permeability in this device, or this changes from \*\* to dark and reverse.

[0021] Drawing 3 shows roughly the cross section of the improved adjustable reflection factor mirror 300 by this invention assembled partially. In this case, a reflective means is high reflecting layer 18A of the prism form mirror

180 by which the laminating was carried out to the surface 131 of one transparent electrode possession side plate 130 of the adjustable permeability device by this invention.

[0022] The cross section of the improved adjustable reflection factor mirror 400 by this invention partially assembled by drawing 4 is shown roughly. In this case, a reflective means is the high reflecting layer 20 on one electrode possession side plate 130 of the adjustable permeability device by this invention, and the laminating of the transparent prism form body 22 is carried out to the surface 101 of the electrode possession side plate 100 of another side of the adjustable permeability device by this invention.

[0023] It sets in one viewpoint and this invention is the (1) and (A) solvent.;

(B) At least one sort of cathode electrochromic compounds accompanied by [ in / among these reduction / in the voltamogram performed using the inert electrode in the room temperature in the above-mentioned solvent, display at least two sorts of chemical reversible reduction waves, and / at least one sort of wavelength of a visible region ] increase of a molar extinction coefficient in the 1st thing;

(C) Display at least two sorts of chemical reversible oxidation waves in the voltamogram performed using the inert electrode in the room temperature in the above-mentioned solvent. The 1st thing is accompanied by increase of a molar extinction coefficient in at least one sort of wavelength of a visible region among these oxidation. When all of at least one sort of anode electrochromic compound; and (D) cathode compounds, and an anode compound are not ionicity in those zero potential equilibrium in the above-mentioned solvent It is the solution which consists of an inactive current conveyance electrolyte and which is used as an adjustable permeability medium in a single partition mold self-elimination type solution phase electrochromic device.

[0024] combining the solution of this invention with material, such as an acrylic sheet material obtained from Lucite L (a registered trademark, LUCITE L) even if, -- others [ gel ] -- or it may be thickened.

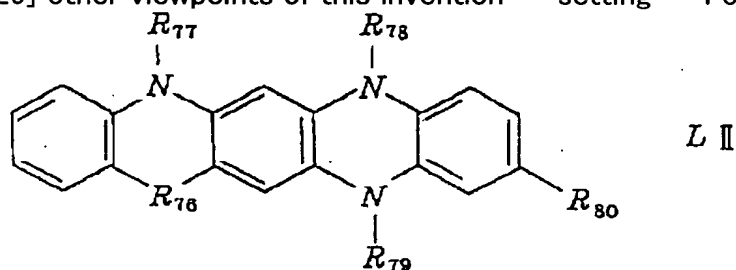
[0025] In other viewpoints of this invention, it is the single partition mold self-elimination type solution phase electrochromic device which consists of a solution of this invention as a medium with the reversible adjustable permeability to light. The solution which has reversible adjustable permeability in the device of this invention may be gelled or thickened.

[0026] This invention offers the adjustable permeability color filter which consists of a single partition mold self-elimination type solution phase device of this invention as an adjustable permeability element in other viewpoints.

[0027] This invention offers the adjustable reflection factor mirror which consists of this invention single partition mold self-elimination type solution phase device as an adjustable permeability element in the viewpoint of further others.

[0028] This invention contains the display device which consists of the adjustable permeability color filter or adjustable reflection factor mirror of this invention as an information conveyance element in the viewpoint of further others.

[0029] other viewpoints of this invention -- setting -- Formula LII -- [Formula 37]



the inside of {type, and R76 -- an oxygen atom or a sulfur atom -- it is -- R80 -- a hydrogen atom or a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) Or it differs. it has 1-6 carbon atoms, respectively -- it is -- or [ that R77, R78, and R79 are the same ] -- The alkyl group, phenyl group (replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration) which have a hydrogen atom and 1-6 carbon atoms, respectively, And the compound of} chosen from benzyl (the phenyl group may be replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration) is included.

[0030] The device in which this invention has reversible adjustable permeability in the viewpoint of further others, It consists of a flat front face and a flat reflective means. The (A) above-mentioned device (i) thermochromic, A medium with the permeability which changes with photochromic one or electro-optics-means reversibly, And before holding the medium parallel at (ii)2 sheet flatness which keeps a gap, is a field and has reversible adjustable permeability among these and reflecting from the above-mentioned reflective means, light passes this behind.;

(B) The angle of a front plane and the plane of a reflective means contains the adjustable reflection factor mirror which is 1-5 degrees.

[0031] In this mirror, remarkable amelioration is obtained by arranging the plane of a reflective means with few



angles to the plane (that is, the light reflected by the mirror from a reflective means advancing into a mirror through this, and coming out of a mirror) of the front face of a mirror. By arranging these planes in this way, the light (namely, light by front residual reflective power) from the mirror outside reflected from the front face of a mirror, without advancing into a mirror is not visible to a mirror user, and the light reflected from the reflective means on the other hand can turn a mirror so that it may be visible to a user. Therefore, the degree of the need of dark-color-izing the medium which does not need to conquer the residual reflective power of the front face of a mirror, therefore has reversible adjustable permeability in order to decrease the reflection (the reflected glare is included) from this kind of mirror is low compared with dark-color-izing considered to be necessity, when a reflective means is parallel to a front face. The distortion of the reflected image which happens when it gazes at the both sides of the light furthermore reflected for the residual reflective power of the front face of a mirror and the light reflected from the reflective means of a mirror is avoided when only the light from a reflective means can be seen.

[0032] Although the medium into which the permeability to a visible ray is reversibly changed by thermochromic, photochromic one, or the electro-optics-means can be used as a medium which all has reversible adjustable permeability in these improved mirrors of this invention The solution (gelled or thickened) according [ a medium ] to this invention is very desirable, And a device with reversible adjustable permeability is a single partition mold self-elimination type solution phase device by this invention. Consist of an parallel side plate which kept the gap by the flatness of two sheets, among those at least one side is transparent (before being reflected from a reflective means, the light reflected in behind by the mirror from a reflective means passes this). Another side is that the high reflecting layer (it acts as a reflective means of a mirror) has adhered to the opposite side of the side in contact with a solution, when not transparent.

[0033] Although it is the same as the thing and the essential target of this invention, the single partition mold self-elimination type solution phase electrochromic device containing the reversible adjustable permeability solution of another kind is known in this technical field. For example, please refer to MANOSU shown above, bread felt, SHIYATSUTSUKU, a SHINSA box, and KUREKAKU's and others specification.

[0034] An exploded view shows the fundamental configuration element of the typical device of this invention to drawing 1 . The flat electrode maintenance side plate or flat walls 100 and 130 of two sheets and a spacer, or a detached core 11 is contained in these elements. In the assembled device, a gap is kept, and a spacer holds walls 100 and 130 in parallel, and encloses space or capacity 12. Capacity 12 is defined in the assembled device with four walls 11A, 11B, 11C, and 11D (in drawing 1 , Walls 11B and 11C are hidden from the visual field) of the electrode layers 10A and 13A of the electrode possession layers 100 and 130, and a layer 11, respectively. In the assembled device, capacity 12 is filled with the solution by this invention (or when leaving room for the expansion accompanying a temperature rise, filled mostly), and this shows reversible adjustable permeability on the occasion of actuation of a device. The solution in capacity 12 touches the both sides of the electrode layers 10A and 13A during the actuation period of a device.

[0035] usually -- and in the assembled device, the electrode layers 10A and 13A and the layers 10 and 13 (the electrode layer has adhered to these) of a solid material are flat, and that of walls 100 and 130 are parallel respectively preferably. In consideration of the possibility of a slight variation produced from few biases, such as the homogeneity of the thickness in a different point (for example, setting to each drawing the layers 11, 10, 10A, 13, and 13A each), and the flexibility of a material, it is in ordinary tolerance and means that it is flat and parallel respectively so that the word of being "flat" and "parallel" may be understood by this technical field in this specification.

[0036] however, capacity 12 was filled with the solution -- as long as it can carry out back (or after filling mostly) seal -- the electrode possession layers 100 and 130 -- not being flat -- moreover, the electrode layer of those insides -- an every place point -- it is -- etc. -- you may not be distance -- \*\* (however, an electrode layer does not contact mutually) -- the solution should be carried out. Furthermore, although the layer 100 and the layer 130 were created from the same material, were equipped with the same thickness and the electrode layer (10A, 13A) of the same material, and are equipped with the same thickness and the solid material layer (10 13) of the same material or will probably essentially be the same in a desirable device, it comes out so and a certain necessity is not. Electrode layers may be a different material and different thickness like a solid material layer.

[0037] In the typical device of this invention, the solid material layers 10 and 13 of walls 100 and 130 will be created from transparent glass with a thickness of 0.05-1cm or transparent plastics, respectively. These are suitable for applying the layer of a conductive material and making the electrode layers 10A and 13A form. However, layers 10 and 13 are transparent and may be created with what kind of material which this is made to fix a conductive material and can form an electrode layer.

[0038] The electrode layers 10A and 13A may be created with what kind of conductive material which is essentially chemical inertness to the solution of reversible adjustable permeability which can be made to adhere to the material of the solid material layers 10 and 13 in the shape of a layer, respectively, and is used for the

device of this invention. The material suitable for an electrode layer is the thin high reflecting layer of materials, such as gold, the tin oxide, ruthenium oxide, stannic-acid cadmium and a thin transparent layer of the tin oxide ("ITO") which carried out the indium dope, a rhodium, or Inconel (Inconel). A desirable thing is ITO. The method of giving a conductive material to the solid material of layers 10 and 13, and forming a suitable electrode layer is learned for this technical field. preferably, as shown in drawing 1 and drawing 2, an electrode layer reaches the whole surface of a solid material layer even on the extension of the solid material layer beyond the outer wall (namely, drawing 1 -- being related -- the wall of the spacer of the opposite side of Walls 11A, 11B, 11C, or 11D) of a cover, capacity 12, a spacer 11, and a spacer 11 -- I will come out. Probably an electrode layer is crossed to the whole surface of the solid material layer which this has fixed preferably, and has the most uniform possible thickness. The thickness of an electrode layer is desirable and this has the low effectiveness below 40-ohm par square more preferably below 100-ohm par square. However, as long as it can create electric contact with an electrode layer and the solution in solution space touches the electrode layer on the occasion of actuation of a device, it is not necessary to spread even on the outside of the spacer held in the condition of wrap necessity not having an electrode layer in the whole solution capacity of the device of this invention, or having left the electrode possession wall of a device. Furthermore, as for a demand, that an electrode layer has uniform thickness or having the low effectiveness below 100-ohm par square are not carried out, either.

[0039] In the device of this invention, the electrode of one side or both may dissociate from the solid material layer, 10 and 13, of each drawing. [ for example, ] About drawing 1, an electrode strip can be arranged in parallel along Sides 11B and 11D instead of the electrode layers 10A and 13A. Or one side of the electrode layers 10A and 13A can also be replaced by the electrode board or strip which has not adhered although it is parallel to the solid material layers 10 or 13 respectively. When the electrode is separated from the solid material layer, an electrode and a solid material layer are created to the solution of this invention with the material which is essentially chemical inertness. In this kind of device, it is suitable for the glass solid material layer, and a rhodium or platinum is suitable as an electrode.

[0040] The device of this invention advances into a device and modulates reversibly the luminous intensity which will come out from now on. Therefore, in the device of this invention, some of one [ at least ] walls [ at least ] of solution space have the transparent permeability of the solution of this invention in solution space to the light of a wavelength range including a part of wavelength range [ at least ] which changes reversibly on the occasion of actuation of this device. Probably, in a typical device, all the fields of the wall of both solution space will be transparent to the light of the whole wavelength of a visible region at least.

[0041] In the desirable adjustable permeability color filter by this invention, the device with reversible adjustable permeability is a device according [ the wall (for example, 100 and 130 of drawing 1) of both solution space ] to transparent this invention to the visible ray of full wave length.

[0042] In order to manufacture the adjustable reflection factor mirror by this invention, a high reflecting layer (for example, silver larer) can be given to one outside (namely, a solution and the opposite side) of the transparent wall of the solution space of the device by this invention. In this case, (except for a reflecting layer) probably, both the walls of solution space will be transparent. Or an adjustable reflection factor mirror can also be manufactured by using the conductive material (for example, a rhodium or Inconel) of high reflective power for one side of an electrode layer which appoints the solution space in a device.

[0043] The transparent wall which defines solution capacity in the device of this invention like a postscript can manufacture the adjustable permeability color filter and adjustable reflection factor mirror to which association, adhesion, or adjustable [ as opposed to / carry out a laminating and / light by the device of this invention ] permeability is given on plates, such as glass or plastics, and a mirror.

[0044] That it is "transparent" means that the light of the full wave length of the range passes without not absorbing at least a part or reflecting it to the light of wavelength set on these specifications. When using the word of being "transparent", without defining a property, this means the transparency to the light of the wavelength of the field which contains visible region (wavelength of 4200-7000A) full wave length at least. The light of the full wave length of the visible region which carries out incidence to this will general still pass the wall with the transparent solution capacity of the device of this invention at least in practice, without not reflecting about 90% or absorbing it.

[0045] On the other hand, more generally in the semantics of this specification, the light of the full wave length of the appointed range reflects the "high reflexivity" surface about 50% at least, without not penetrating about 70% or absorbing it. When using without defining a property, it comes out so to the light of the full wave length of a visible region at least, and the surface of "high reflexivity" is a certain thing.

[0046] The spacer expressed with 11 in each drawing is insulation, and is created from combination with a sealant, for example, an epoxy resin, silicone, rubber cement, low melting glass, specific plastics, paraffin wax, etc. a spacer material, for example, a small glass bead, and a nylon monofilament, a Mylar (MYLAR, registered trademark) strip, polystyrene beads, etc. As mentioned above, a spacer is the thing of uniform thickness substantially preferably, therefore the wall of two sheets which appoints the solution space of a device is held in

parallel essentially mutually. Although roughly shown in drawing 1 , they are whether the common-law marriages 11A, 11B, 11C, and 11D of a spacer and the rim of the opposite side of a common-law marriage are curving in fact, and a rude edge. This curvature nature or roughness will become clear from the assembly format of a common device. That is, the strip of the mixture (high viscosity) of a sealant and a spacer material is put on the surroundings of the fixed field inside one wall of a device (namely, electrode layer possession side), and subsequently to the inside of the 1st wall, in the condition that the wall of that inside (namely, electrode possession side) faces the wall of another side of a device each other, it is assembled by the strip by \*\*\*\*\* until both walls contact this separation means (spacer). The superfluous sealant of a strip is pressed out by this pressure from a strip, and the rim and common-law marriage of a strip curve, or become an ununiformity. the device of this invention -- setting -- a separation means (spacer) -- the inside (namely, electrode possession side) of a wall -- about -- it maintains at the condition of having separated 0.0025--about 0.05cm. A desirable spacer is the combination of a glass bead and an epoxy resin system sealant.

[0047] It can connect with DC power source, or can connect, and, thereby, the electrode of the device of this invention can give potential to the solution of a device again inter-electrode. The desirable array format for connecting an electrode to a power supply is shown in the device roughly shown in drawing 2 . In this format, in order to give the strip which each electrode side exposed, the electrode possession wall of two sheets was inquired of the longitudinal direction from solution space, and is shifted to the opposite direction in parallel with this. In order [ of the length which the strip exposed to each of these exposed strips ] to contact this and an electric target along with the whole mostly, the strip or line of a conductive strip or a line, for example, copper, aluminum, or silver is pasted up. On the whole, one side of this kind of strip and 16 are shown to drawing 2 by the cross section at drawing 3 and drawing 4 . Only the lead wire of the strip of another side of the device of drawing 2 or production 15A is visible to drawing 2 . A strip 15 is visible to drawing 3 and drawing 4 in a cross section. The strip 15 as well as the strip 16 adhering to electrode layer 13A has essentially adhered to electrode layer 10A along with the overhang overall length of an electrode layer. Although each means (for example, a clamp stop, soldering, or immobilization by electroconductive glue) known for this technical field is employable in order to fix a line or a strip, where an electrode surface and an electric target are contacted, a desirable means is using a conductive epoxy resin, for example, a standard silver epoxy resin. The strip or line adhering to an electrode surface contains lead wire or a production exceeding the end of an electrode side, as 15A and 16A show to drawing 2 . Connection with a suitable power supply is made by the standard electrical connection from a power supply to these lead wire or productions.

[0048] The assembly of the device of this invention can be performed so that I may be understood by this technical field. Please refer to the specification of the above-shown of MANOSU. A desirable device assembly method is as follows.

[0049] The strip of the spacer material which consist of what mixed separation material ( for example, glass bead) with the sealant ( for example, insulating epoxy resin) be make to adhere to one surface of a device ( set for the desirable example which the layer of the conductive material with which the surface of; device be a flat solid material piece, for example, glass, and be use for the electrode surface as an electrode at this have adhere or paste up), and this enclose the cross section field for solution capacity of a desirable size and a Subsequently, solution capacity is formed by putting the surface of another side of a device in the condition that the electrode layer of each surface faces mutually, on the strip of a spacer material. Subsequently, a pressure is put on the 2 surfaces, and it is made to approach mutually until these will be substantially separated by only the separation material of a spacer. When making the solution used with a device thicken with combination with a thickener (for example, acrylic sheet material led from Lucite L), before giving the 2nd wall to the whole field surrounded by the spacer on the 1st wall in the solution of the thickener in a volatile solvent (for example, dichloroethane, an acetone, or a methyl ethyl ketone), it is convenient spreading or to carry out a spray and to evaporate a solvent. after an assembly production process -- and in being required before solution restoration, the web material of a spacer is stiffened and it makes with inactive in a solution. This hardening is required, when the solvent of a solution is propylene carbonate and a sealant is an insulating epoxy resin.

[0050] The configuration of the solution capacity seen through the electrode possession wall in the cross section is not restrained by a square or the rectangle. This may be the form of circular, an ellipse, a polygon, an alphabetic character, or a numeric character, or what kind of configuration for which it wishes.

[0051] in the device assembled by one wall of the device of this invention, two small holes can open near upper part \*\*\*\*\* of solution capacity ( drawing 1 -- one side -- near wall 11A -- another side -- the wall 11 neighborhood). (before assembling) It lets these holes pass to a device, among those a solution is introduced from one side, and it is filled up with the solution of this invention by making air escape from another side. The seal of these two holes is first carried out with inactive common thermoplastics to a solution after restoration, and, subsequently a seal is carried out by the sealant (for example, insulating epoxy resin).

[0052] Subsequently, a conductive line or a conductive strip (usually copper strip) is usually pasted up on the outcrop of a two-electrodes side with a conductive epoxy resin (for example, standard silver epoxy resin).

Finally, the seal of a line or the strip (except for the lead wire or the production which forms it, a power supply, and a contact) is carried out like the perimeter (namely, the edge containing a spacer or the outside of the side) of a device using the sealant used for the spacer.

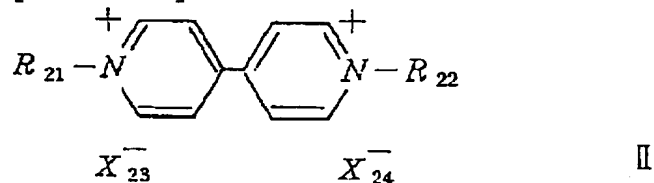
[0053] Each mixture of the compound with which it is known over the temperature requirement where the solution of this invention is used as a medium which has reversible adjustable permeability in the device of this invention that it is liquefied and useful as a solvent in the electrochemical technique field, or a compound can be used for the solvent in the solution of this invention. A solvent is a liquid at at least 20–27 degrees C (namely, room temperature), since the device of this invention is operated triggered by the facilities at the time of adjusting a solution in fact over the fixed temperature requirement which usually includes a room temperature. Furthermore, it is desirable for the solvent of the solution of this invention to receive electrolysis during storage of a device or ordinary actuation for the stability of the device of this invention, or not to participate in other irreversible chemical reactions. It is water, a methanol, ethanol, an acetonitrile, N.N-dimethylformamide, dimethyl sulfoxide, an acetone, a methyl ethyl ketone, a cyclopentane, and the cyclic ester that were suitable as a solvent, and propylene carbonate, ethylene carbonate, beta propiolactone, beta-butyrolactone, gamma-butyrolactone, gamma-valerolactone, delta-valerolactones, or such homogeneous (namely, single phase) mixture are contained in these. As for a solvent, it is substantially desirable that it is anhydrous excluding dissolved oxygen except water. Desirable solvents are cyclic ester or those combination. A very desirable thing is propylene carbonate.

[0054] At least one sort of cathode electrochromic compounds are more ordinarily contained in the solution of this invention by the concentration of Abbreviation 0.01–0.1M from 10–4M to the solubility at least in 25 degrees C. Measuring this with a standard BORUTAMO graphic method at a room temperature in an inert electrode in the solvent of a solution, the 1st thing follows increase of the absorbancy index of a cathode compound on at least two sorts of chemistry targets in at least one sort of wavelength of a visible region including a reversible reduction wave ( namely, the thing understand in the field of electrochemistry technology; you may not necessarily are reversibility dynamically) among these reduction. Furthermore, at least one sort of anode compounds are more ordinarily contained in the solution of this invention by the concentration of Abbreviation 0.01–0.1M from 10–4M to the solubility by 25 degrees C. this -- the inside of the solvent of a solution -- an inert electrode -- setting -- a room temperature -- standard Volta MOGURAFU -- law -- measuring -- at least two sorts -- the 1st thing is chemically accompanied by increase of an absorbancy index in at least one sort of wavelength of a visible region including a reversible oxidation wave (what is understood in the field of electrochemistry technology) among these oxidation.

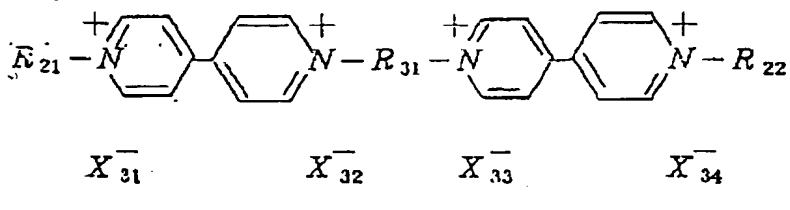
[0055] Usually, if the potential difference is given to the inter-electrode solution of the device of this invention, it will be meant that a solution changes from \*\* to dark, or a color changes. therefore, the 1st chemical reversible reduction of the cathode electrochromic compound used for the solution of this invention or the 1st chemical reversible oxidation of an anode electrochromic compound -- following -- the inside of the solvent of a solution -- a room temperature -- setting -- at least one sort of wavelength of a visible region -- at least -- about 102- at least -- about 103 -- it is desirable for increase of the absorbancy index of the factor of cm-1M-1 to take place.

[0056] The compound of Formula II known [ compound / suitable for the solution of this invention / cathode electrochromic ] (biotechnology ROGEN)

[Formula 38]



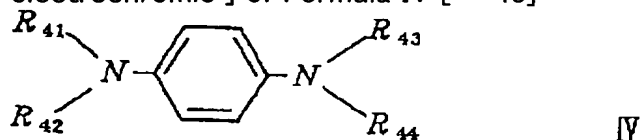
Whether R21 and R22 are the same among [type Or the alkyl group which differ and has 1–10 carbon atoms, respectively, a phenyl group (cyano \*\* the location of arbitration -- setting -- chloride, bromide, and iodide --) Or you may be replaced by the alkyl group which has 1–4 carbon atoms. and benzyl (a phenyl group -- the location of arbitration -- setting -- chloride and bromide --) It is chosen [ that you may be replaced by the alkyl group which has iodide and cyano \*\* or 1–4 carbon atoms and ], and whether;X-23 and X-24 are the same Or a difference, ]; chosen from chloride, bromide, iodide, BF-4, PF-6, ASF-6, ClO-4, and NO-3, respectively, and the compound of Formula III [\*\* 39]



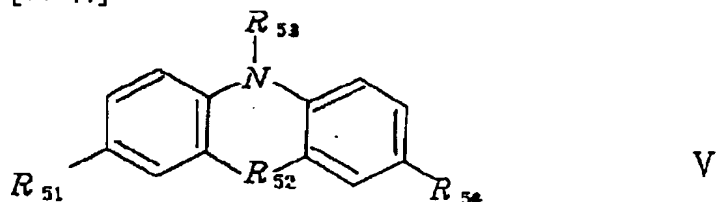
(-- or [ the inside of a formula, and / that R21 and R22 are the same ] -- or -- differing -- the compound of point \*\*\*\*\* II -- setting -- R31 -- 1- -- or [ that are the alkylene group which has ten carbon atoms, and X-31, X-32, X-33, and X-34 are the same ] -- or it differs and is chosen out of chloride, bromide, iodide, BF-4, PF6, AsF6, ClO-4,

[0057] All the anions of the desirable compound of Formulas II and III are the same, and it is the thing of ClO-4 or BF-4. A very desirable thing is BF-4. the desirable cation of the compound of Formula II -- R21 and R22 -- the same -- benzyl, phenyl, or n-heptyl -- it is benzyl very preferably. R31 is -(CH2) 4-, as for the very desirable cation of the compound of Formula III, R21 and R22 are the same, and it is the thing (namely, tetramethylenebis [4-(1-benzyl pyridine-4'-IRU) pyridinium]) of benzyl.

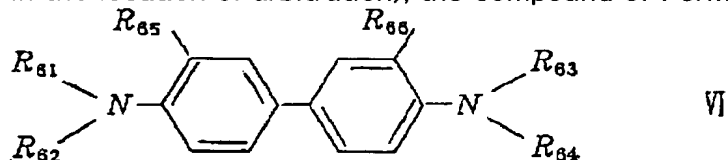
[0058] The compound of the known [ compound / suitable for the solution of this invention / anode electrochromic ] of Formula IV [\*\* 40]



Among [type whether R41, R42, R43, and R44 are the same Or a difference, the full kill radical which has a hydrogen atom and 1-10 carbon atoms, respectively, and a phenyl group (the location of arbitration -- setting -- chloride --) You may be replaced by the alkyl group which has bromide, iodide, and cyano \*\* or 1-4 carbon atoms. And] chosen from benzyl (the phenyl group may be replaced by the alkyl group which has chloride, bromide, iodide, and cyano \*\* or 1-4 carbon atoms in the location of arbitration); the compound of Formula V [\*\* 41]



or [ the inside of [type, and / that R51 and R54 are the same ] -- or -- differing -- respectively -- a hydrogen atom and a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) It is chosen out of having 1-6 carbon atoms, respectively.;R52 An oxygen atom, Whether it is a sulfur atom or NR55, and R55 is the same as that of R53 here Or a difference, The alkyl group in which both R55 and R53 have a hydrogen atom and 1-10 carbon atoms, a phenyl group (cyano \*\* the location of arbitration -- setting -- chloride, bromide, and iodide --) Or you may be replaced by the alkyl group which has 1-4 carbon atoms. Or] chosen from benzyl (the phenyl group may be replaced by the alkyl group which has chloride, bromide, iodide, and cyano \*\* or 1-4 carbon atoms in the location of arbitration); the compound of Formula VI [\*\* 42]



Or it differs. or [ the inside of a formula, and / that R61, R62, R63, and R64 are the same ] -- It is chosen out of the alkyl group or phenyl group which has 1-10 carbon atoms, respectively, and whether;R65 and R66 are the same Or a difference, It is chosen out of the alkyl group which has a hydrogen atom or 1-10 carbon atoms, respectively. However, as for R65 and R66, when both sides are hydrogen atoms, or both sides are alkyl groups and R65 and R66 are [ both sides ] hydrogen atoms, one or more [ of R61 and R62 ] is a hydrogen atom, and one or more [ of R63 and R64 ] is a hydrogen atom.

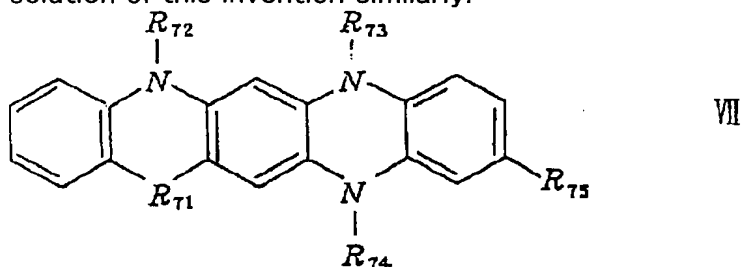
The compound of Formula VIII (tetrathiafulvalene)

[Formula 43]



\*\*\*\* rare \*\*.

[0059] It is the new molecular entity [\*\* 44] of Formula VII which was suitable as an anode compound in the solution of this invention similarly.

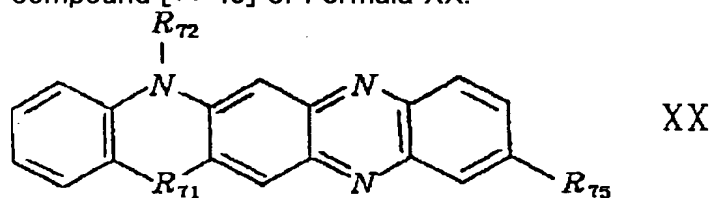


the inside of [type, and R71 -- an oxygen atom or a sulfur atom -- it is -- R75 -- a hydrogen atom or a dialkylamino radical (or it differs or [ that an alkyl group is the same ] --) it is chosen out of the alkyl group which has 1-6 carbon atoms, respectively -- it is -- or [ that R72, R73, and R74 are the same ] -- or -- differing -- respectively -- a hydrogen atom -- The alkyl group which has 1-6 carbon atoms, a phenyl group (replaced by the alkyl group which has 1-6 carbon atoms in the location of arbitration), and benzyl (a phenyl group is set in the location of arbitration) It is] chosen [ that you may be replaced by the alkyl group which has 1-6 carbon atoms, and ].

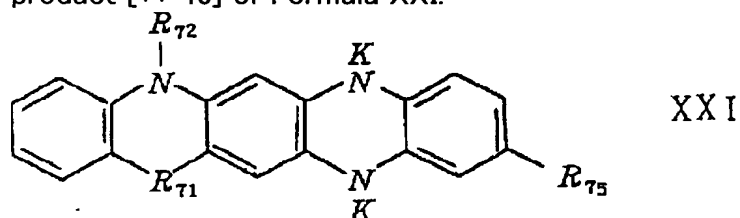
[0060] Among the compounds of Formula VII, R71 is an oxygen atom, R75 is a hydrogen atom, and all of R72, R73, and R74 of a very desirable thing are methyl groups.

[0061] A desirable thing is the compound of Formulas IV and V among the anode electrochromic compounds used for the solution of this invention. Formula IV and \*\* which R41, R42, R43, and R44 are the same as for a more desirable thing, and are a methyl group or a phenyl group -- and R51 and R54 are hydrogen atoms, and R52 is equal to N-R53, and it is the thing of the formula V whose R53 is a methyl group or a phenyl group. A very desirable thing is N, N, N', and N'-tetramethyl. -- It is 1 and 4-phenylenediamine and 5, and 10-dihydro. -- They are 5 and 10-dimethyl phenazine.

[0062] Manufacture of the new molecular entity of the formula VII of this invention follows the known method by Guillemin and the dee trick (79 a journal OBU American chemical society, 6178 (1957)), and is the known compound [\*\* 45] of Formula XX.



It leaves (R71, R72, and R73 set about Compound VII previously among a formula), and is the potassium addition product [\*\* 46] of Formula XXI.



The purpose product is obtained, after making this addition product react with the mixture of the compound of formula R73I and R74I (R73 and R74 may set about Formula VII first among a formula, and it may be the same) and crystallizing it subsequently, nothing and. This synthesis method is shown in Example 11 about desirable N, N', and N''-trimethyl TORIFENA gin oxazine.

[0063] When a cathode electrochromic compound and the anode electrochromic compound of the solution of this invention all are not ionicity in those zero potential equilibrium in a solution, an inactive current conveyance electrolyte is contained, or when that is not right, a current conveyance electrolyte with this inactive kind may be contained by request. While current is carried over an inter-electrode solution while the device of this invention is operating ordinarily, and a device is stored or operating ordinarily, an inactive current conveyance electrolyte receives electrolysis, or performs other material in a device, and other irreversible chemical reactions, and does not spoil the stability of a device.

[0064] The current conveyance electrolyte inactive [ in the solution of this invention ] will consist of being [ they / any of the material (by the way, called a "supporting electrolyte" by this technical field) with which it is

known that it is suitable as an inactive current conveyance electrolyte by this technical field ] combination. An alkali-metal salt, tetra-alkylene ammonium salt, an aluminum chloride, and the aluminium bromide are contained in this kind of material. a thing desirable as a cation in a current conveyance electrolyte inactive [ in the solution of this invention ] -- a lithium, sodium, and tetra-alkylammonium (an alkyl group is the same) -- it is --; -- a very desirable thing is tetra--n-butyl ammonium. a thing desirable as a current conveyance electrolyte inactive [ in the solution of this invention ] -- chloride, BF<sub>4</sub>-, and ClO<sub>4</sub>- -- it is --; -- a very desirable thing is BF<sub>4</sub>-. The concentration will be 0.005–2M at 25 degrees C, when an inactive current conveyance electrolyte exists in a solution. It will be 0.05–0.5M at 25 degrees C very preferably.

[0065] The solution of this invention is for using it as an adjustable permeability medium in a single partition mold self-elimination type solution phase electrochromic device. Since a device is a "solution phase", about the device which operates over a fixed temperature requirement with the potential which does not exceed the fixed maximum given to the solution As for the concentration of the material in a solution, cathode electrochromic material (one sort or two sorts or more) is returned in a cathode during actuation of zero potential equilibrium and a device. A deposit of the material from a solution must not take place in the both sides at the time of anode electrochromic material (one sort or two sorts or more) oxidizing in an anode. Generally, if all material exists in a solution by concentration lower than those solubility in all the temperature of the use range meant by zero potential equilibrium, the deposit will not take place during actuation of the device which contains this solution as a medium with reversible adjustable permeability.

[0066] or [ that the inter-electrode potential difference of a device falls with the "self-elimination (self-erasing)" nature of the device of this invention ] -- or after removing, the permeability of the solution in a device means increasing even to a value characteristic of the new potential difference without the necessity of reversing the polarity of an electrode. the "self-elimination" nature of the device of this invention is obtained by the oxidized anode compound and the returned cathode compound reacting spontaneously (probably -- the bottom of a diffusion limit), and giving the anode compound and cathode compound of zero potential equilibrium, respectively.

[0067] In case the device of this invention is actually used, it is important that reduction of the permeability of the solution of the device which happens when the inter-electrode potential difference increases, and increase of the permeability of the solution of the device which happens with self-elimination take place promptly both enough. As for reduction and increase of permeability, generally, happening as promptly as possible is advantageous. The cathode compound and anode compound with which this invention suits aforementioned Volta MOGURAFU and the criteria of colorimetry were not combined in the solution. When both sides use for a single partition mold solution phase electrochromic device the cathode compound and anode compound with which an absorbance increases in the wavelength range made into the purpose with reduction and oxidation, respectively The speed of a permeability fall turns into speed which can be received for commercial use of this kind of device. Not being lost, so that the speed of the permeability increase by self-elimination which became possible with the solution phase property of this device at that time spoils commercial use is not recognized by this technical field.

[0068] Furthermore, as for the solution in a device, in the both sides under the period when the device is not operating, and circulation, a stable thing is important for the practicality of the device of this invention (namely, when the inter-electrode potential of a device circulates from zero or a low value to the high value and the value of a basis and is, as a result, changing reversibly between a value with the high permeability of the solution in a device, and low values). Lack of stability is shown by when the permeability of the white light which passes a device (the solution in it is included), or the light of wavelength from which an absorbance changes with these devices increases in case it is in equilibrium in case there is no potential difference of zero potential equilibrium, i.e., a device, inter-electrode in a solution.

[0069] The problem which has barred commercial use of a single partition mold self-elimination type solution phase electrochromic device was lacking the stability of the adjustable permeability solution used for them. Although the reason of this instability of the device by the advanced technology is not completely clear, both the anode compound used into the solution of the advanced technology, and cathode both [ one side or ] are considered to relate to it being reactivity in the condition of having been oxidized and returned, respectively at the material and altitude of a, chemically unstable thing, and a solvent and others. This invention solved this problem by the knowledge that the properties of the solution of this invention are remarkable and, unexpectedly high stability, especially the stability over circulation, when the cathode and anode electrochromic compound which meet the aforementioned Volta MOGURAFU criteria were used.

[0070] Rising further was found out, when the oxygen in a solution and a solvent were nonaqueous nature and the stability of the solution of this invention stopped the concentration of water to the minimum. Therefore, before the device of this invention is filled up with a solution, the flash of it is carried out by the inert gas of the nitrogen dried by request (preferably), and others. When the oxygen in the solvent used for preparation of a solution and a solute and a solvent are nonaqueous nature, in order to reduce the concentration of water, a



device is filled up with a solution, and the standard method is adopted, in order to suppress to the minimum that a solution is polluted with oxygen and water before carrying out the seal of the device with which it filled up. For example, desiccation nitrogen can be blown into the solution before restoration in order to reduce an oxygen density. Before using a solvent for preparation of a solution, it can process by passing a drying-agent (for example, activated alumina) top, and contamination by water can be decreased. Furthermore, before using a solute (electrochromic compound; inactive current conveyance electrolyte) for preparation of a solution, you may make it dry by heating at about 110 degrees C. Or before filling up a device with the prepared solution, it can also flow in a drying agent (for example, activated alumina).

[0071] the inside of the above-mentioned treatment taken in order to reduce the concentration of the oxygen in the solution of this invention, and water -- either -- in respect of except, the solution of this invention is prepared by the standard method by usually only dissolving the solute of the amount suitable for attaining the purpose concentration at a room temperature into a solvent.

[0072] A fixed advantage is acquired by using the solution which set to the device of this invention, and was thickened or gelled as a reversible adjustable permeability medium. Still as follows, the above and when a single partition mold self-elimination type solution phase electrochromic device carried out prolonged continuation actuation, it was found out in relation to this invention about this device that segregation is a problem. By decreasing the element of the segregation resulting from a free convection, the noteworthiness of segregation decreases by making the solution of this invention gel or thicken.

[0073] Other advantages realized by using for the device of this invention the solution gelled or thickened relate to facilities and safety. For example, the solution which must open a device by one destruction and others of a transparent side plate and which was case [ the solution ], gelled or thickened flows out gradually far rather than what is not being gelled or thickened, therefore the clarification ease of carrying out of a solution increases, and the danger that people will contact the harmful material which may exist in a solution will decrease. In the device which the element of a transparent side plate and transparent others may crush on the occasion of failure, the solution gelled or thickened has the orientation to hold a fragment on that spot, and the danger of the trauma which may happen when the device of \*\* is distracted by this decreases.

[0074] On these specifications, "it thickening (thicken)" and the word of "gelling (gel)" are used as a synonym, and mean a viscosity rise of the solution produced by aligning a solution with specific material regardless of whether gel true in this process is formed. Without reacting with the solvent in a solution, an inactive current conveyance electrolyte, an anode, or a cathode compound, and forming covalent bond, each material which is made to thicken a solution and deals in it can be used in order to make the solution of this invention thicken or gel. What is necessary is just to only align the object for thickening or the material for gelation of the amount of the purposes with a solution, just before filling up a device when there is sufficient time amount to be filled up before a solution becomes hyperviscosity too much. Or before introducing a solution, the object for thickening and the material for gelation of the amount of the purposes can be inserted in a device behind, and a solution and mixture can also be obtained in a site in the solution space of a device. An example (a thickener is introduced in front of a solution) of this method is shown in Example 10.

[0075] The concentration of the object for thickening or the material for gelation used in order to prepare this invention solution thickened or gelled will change according to the factor of a large number understood by this contractor. The object for thickening or the material for gelation to be used, the solvent to be used, and the viscosity made into the purpose of a solution thickened or gelled are contained in these factors. the constituent obtained by dissolving the propylene carbonate which is a desirable solvent, and a thickener desirable to this solvent, i.e., the acrylic sheet material marketed by the registered trademark of Lucite L, in an organic solvent (for example, an acetone, a methyl ethyl ketone, or dichloroethane) -- the concentration of the thickener in a solution -- about 3-- about 30% (W/W) -- desirable -- about 5-- about 25% (W/W) -- more -- desirable -- about 7-- it is about 15% (W/W).

[0076] Other specific thickeners which can be used in order to thicken or gel the light solution from a book on the specifications of MANOSU shown above are listed. It was admitted that the constituent obtained by dissolving the acrylic sheet material marketed by trademark Plexiglas (PLEXIGLAS) about this invention using a propylene carbonate solvent in an organic solvent (for example, an acetone, a methyl ethyl ketone, or dichloroethane) could also be used for thickening.

[0077] It mixes with a solvent (Lucite L), for example, the acrylic sheet material of marketing of dichloroethane (mixture of 1,2-dichloroethane, 1, and 1-dichloroethane or 1, 1 and 1, and 2 isomer), and a desirable thickener separates the obtained solution with residue, and is obtained by finally evaporating a solvent. The residue left behind after a solvent evaporates is "the thickener which consists of an acrylic sheet material."

[0078] Since the device of this invention using a propylene carbonate solution as a medium in which using this desirable thickener unexpectedly has reversible adjustable permeability about this invention was constituted, having been remarkably suitable very conveniently was admitted. A spray is applied or carried out. as shown in Example 10, this thing convenient and suitable the solution of this thickener only to the electrode possession



side of the wall of a device before device assembly. Subsequently, the thickener inside that the thickener of a constant rate can be inserted in a device by evaporating a solvent and a device is automatically incorporated by the propylene carbonate solution, after the assembled device is filled up with a solution by the conventional method, and it is based on making this thicken.

[0079] The very desirable property that the solution of this invention thickened with the thickener which consists of a desirable acrylic sheet material found out in relation to this invention is unexpected is not increasing more nearly intentionally than the time amount which coloring of the same device takes except the point that the time amount which coloring of a device which used this kind of solution as a reversible adjustable permeability medium takes is not included for a thickener in a solution. Therefore, it realizes about this kind of thickener, without having effect for the advantage of quick coloring of the device using the non-thickening solution of this invention as a reversible adjustable permeability medium with the aforementioned disadvantageous advantage by using the thickened solution as a reversible adjustable permeability medium in the device of this invention.

[0080] In order to make it operate, the device of this invention is connected to the power supply which can establish the polar fixed potential difference to inter-electrode [ of a device ]. If drawing 1 and drawing 2 are described, this connection will let the conductive line which fixed in the electrode layer of the wall of a device, or the lead wire 15A and 16A of a strip pass, and it will be performed so that it may be in an electrode layer and a conductive contact condition. A power supply may be AC known by this technical field, or any of a DC power supply. However, in the case of an AC power, a controlling element (for example, diode) is arranged between a power supply and each electrode of a device, and it is guaranteed that the polarity of the inter-electrode potential difference does not change with fluctuation of the electrode of the potential from a power supply. Suitable DC power supplies are a battery, for example, the cell for automobiles, and a dry cell. The power sent to each electrode of the device from a cell is controlled by one of the means known for this technical field, and it is made for the potential given to the inter-electrode solution of a device not to exceed the potential difference from which irreversible reaction (for example, electrolysis of a solvent, reduction of an inactive current conveyance electrolyte or oxidation, the unimolecular decomposition reaction of an electrochromic compound, etc.) occurs. In order to use the gray-scale organization potency of the device of this invention, control of the power preferably sent to the electrode of a device is kept constant from about 0.1V in any potentials which he can fluctuate over the range to potential [ a little ] lower than the potential from which irreversible reaction occurs in a significant degree, however this range wishes in a device in potential. Probably, a switching means by which the inter-electrode potential of a device can be reduced even to zero by open circuit formation or the short circuit also accompanies the power supply. When it is the specification by which an additional self-elimination speed is attained by giving about potential a short period of time (for example, for about 0.5 – about 5 seconds) (polarity is reversed with the thing of a permeability enlargement stage throughout), a switching means can also include the means for performing this inversion in an electrode. The means and the switching means of controlling the potential difference given to an electrode can be operated with either hand control or an automatic.

[0081] In order to oxidize and return the electrochromic compound in the solution of this invention and to reduce the permeability of a solution by this, the inter-electrode potential difference must be so high that it is enough to pass current over an inter-electrode solution. In current, a sink and the solution of this invention are usually suitable for the potential difference of abbreviation 0.3 – about 0.5 V, dark-color-izing or although it begins to carry out color change.

[0082] The degree dark-color-ized by the steady state in the specific device of this invention will be based on the inter-electrode potential difference. The device of this invention is useful as a "gray-scale" device because of this property.

[0083] It will depend for the highest potential which can be given to inter-electrode [ of a device ], without spoiling the stability of a solution on many factors, for example, the potential to which electrolysis of a solvent takes place, and the potential in which the decomposition reaction of an electrochromic compound occurs so that I may be understood by this contractor. The device of this invention whose solvent in a solution is water will be operated by about 1.4v or less, in order to avoid electrolysis of water generally. The device of this invention using a cyclic ether system solvent can operate it, being able to apply the high potential difference which attains to about 4 V by the case at a solution layer. However, the potential generally given to a solution layer in the device of this invention is kept at less than [ 2V ].

[0084] By the steady state to which fixed voltage was given, a cathode electrochromic compound is continuously returned to the solution layer of the device of this invention in an electrode. To that an anode electrochromic compound oxidizes and electrochemical oxidation and reduction take place and coincidence, at and this speed Reverse oxidation of the returned cathode compound being carried out by the reaction of the returned cathode compound and the oxidized anode compound, and reverse reduction of the oxidized anode compound being carried out, and returning to those zero potential equilibrium by it will be understood by this

contractor. It depends for the speed from which a steady state is attained where fixed potential is given to the solution of a device on the current which passes along a solution in the potential. It is not thought that current is the variable which generally became independent on the occasion of actuation of a device. It is because these are dependent on the conductivity (this is dependent on the presentation of a solution including the presentation of an inactive current conveyance electrolyte) of other factors which change independently, for example, the solution in a device, and the potential given to a solution. However, generally the current which flows during ordinary device actuation is in the range of 0.1–20mA per cm<sup>2</sup> of the cathode area in contact with a solution layer, or anode area.

[0085] As mentioned above, the problem found out in relation to this invention is that segregation happens in the single partition mold self-elimination type solution phase electrochromic device continuously operated over the long period of time for about 20 minutes or more (that is, maintained at non-zero potential). It is thought that this segregation is the same as the segregation which encounters on the occasion of actuation of a large-scale electrochemical tub. For example, the segregation accepted in the device of this invention contains the component by the natural convection current produced with different partial density (one side is higher than apparent solution density, and one side is low) in the perimeter of the component by the electrified electrochromic compound shifting in the electrochemical electric potential gradient in the solution of a device, the oxidized anode molecule, and the returned cathode molecule.

[0086] It is desirable to avoid segregation in the device of this invention. It is because troublesome color separation happens to the solution layer of the device of this invention and the self-erasing speed of a device falls by this.

[0087] As mentioned above, in the device of this invention, it is using the solution which used as the medium of segregation in which law's has reversible adjustable permeability on the other hand in order to decrease a free convection component at least, and this invention's thickened or gelled.

[0088] Moreover, in relation to this invention, it was found out that segregation can be substantially removed by the following in the device of this invention.

[0089] (a) It is the minimum of the density range which can be received in order to attain the permeability fall of solutions for the use of the schedule for which the device is used with the enough concentration of the (i) cathode and an anode electrochromic compound, and the concentration of (ii) current conveyance electrolyte uses twice [ at least ] and a desirable at least 10 times higher solution for a device rather than the total concentration of an anode compound, or the total concentration of a cathode compound. And when (b) drawing 2 is described, either a conductive strip or a line (about 16 and a strip, or a wire (not shown), lead-wire 15A is a production) is in a location (namely, the one distant from surface of the earth) higher than another side. In order to reduce the permeability of the solution in a device below to a zero potential balanced value or to hold to this, in case device potential is given the conductive strip or line of a location of the higher one is put on the potential of the higher one (therefore, the electrode which this has connected is an anode) — a device is made to arrange like

[0090] For example, when it arranges as mentioned above, the device of this invention which contains the solution indicated for Example 12 as an adjustable permeability medium does not show segregation, so that it can be accepted, even if it makes it operate continuously by 1.0V for 24 hours.

[0091] This invention relates to the adjustable reflective mirror improved in the last viewpoint. The desirable gestalt is roughly shown in mirrors 300 and 400 at drawing 3 and drawing 4 , respectively. The amelioration in these mirrors is obtained as mentioned above by arranging a flat reflective means (it being shown in drawing 3 as 18A, and shown in drawing 4 as 20) with few angles to the front face ( drawing 4 being shown as the surface 221 of the piece 22 of a prism form of a mirror 400 in drawing 3 as the solid material layer of the wall 100 of a mirror 300, and the surface 101 of 10) where a mirror is flat. The front face of a mirror is the surface which light passes this, and advances and comes out of a mirror through this.

[0092] These mirrors of this invention contain the reversible adjustable permeability device which passes this behind, before light is reflected from a reflective means.

[0093] A reversible adjustable permeability device features the light of wavelength into which the reflection factor of a mirror is changed at least, and the surface in which the parallel gap was preferably kept by the transparent flatness of two sheets to the light of all the wavelength of a visible region at least, and the medium from which an absorbance changes with thermochromic, photochromic one, or electron optics—means reversibly is arranged among these. About the mirror 400 shown in the mirror 300 shown in drawing 3 , and drawing 4 , these surfaces are the surface 101 of the solid material layer 10, and the surface 131 of the solid material layer 13.

[0094] Although a reversible adjustable permeability device (it has the surfaces 101 and 131) is an equal electrochromic device in the mirrors 300 and 400 of drawing 3 and drawing 4 as substantially as the device of this invention shown in drawing 2 , it is not limited only to the mirror by which this invention was improved containing the single partition mold self-elimination type solution phase electrochromic device of this invention

as a reversible adjustable permeability device, respectively. Each device into which permeability is changed by thermochromic, photochromic one, or the electron optics—means can be used in order to change the reflection factor of the mirror by which this invention was improved. However, in this kind of device, an adjustable permeability medium is held between the surfaces in which the parallel gap was kept by the transparent flatness of two sheets to the light of the wavelength of the schedule into which the reflection factor of a mirror is changed at least. The electron optics device of many molds suitable for this purpose is known (for example, a liquid crystal device, a bipolar suspension device, an electromotive type device, a 2 partition mold electrochromic device (for example, thing reported by KISA)).

[0095] In one gestalt (shown by the mirror 300 of drawing 3 ) of the adjustable reflection factor mirror by which this invention was improved, the laminating of the prism form mirror 180 is carried out to the surface 131 of a reversible adjustable permeability device by the transparent charge of a laminated wood (shown by the layer 19). A prism form mirror is a general prism form mirror used for the reflector glass of an automobile. A prism form mirror consists of layer 18A of the high reflexivity material (for example, silver) made to essentially adhere on the surface of a solid material with standard technology in the piece 18 of the transparent prism form of a solid material (for example, glass or transparent plastics), and the field of mirror processing technology. It is reflected in the direction of even if the high rate (desirable at least about 80%) of light which passed the solid material by this and reached the reflective material layer passes along a solid material layer. The high reflector of a prism form mirror is a wrap about all cross sections (although shown to drawing 2 by 14, not shown in the cross section of drawing 3 ), even if there are few reversible adjustable permeability fields of the reversible adjustable permeability device of the improved mirror. High reflecting layer 18A is a reflective means of a mirror by which this invention was improved.

[0096] The layer of the charge of a laminated wood does not exist in the adjustable reflection factor mirror (similar to what was shown in drawing 3 ) by which this invention of other molds was improved. Instead, the surface of the prism form mirror which is not covered with a high reflecting layer is covered with a conductive layer (it functions as an electrode of a reversible adjustable permeability device), and a prism form mirror replaces by the wall 130 as one wall of this device with an electrode layer.

[0097] In the case of the adjustable reflection factor mirror (shown by the mirror 400 of drawing 4 ) by which this invention of the mold of further others was improved A reflective means is the layer 20 of the high reflexivity material (for example, silver) made to adhere to the surface 131 of a reversible adjustable permeability device by one in the mirror manufacture industry of the standard methods. It is reflected in the direction of even if the high rate (desirable at least about 70%) of light which passes a reversible adjustable permeability device by this, and reaches a reflexivity material passes along the surface 131. Furthermore, in the case of the mirror of the improved mold which is shown by the mirror 400 of drawing 4 , it is parallel to the surface 131 and the laminating of the surface of the reversible adjustable permeability device shown by 101 in which the gap was kept from now on is carried out to the transparent piece 22 of a prism form made from a solid material (for example, glass or transparent plastics) by the transparent charge of a laminated wood (shown by 21). One surface 221 of the piece of a prism form is a front face of an amelioration mirror, advances into a mirror through this, and, as for the light reflected by the reflective ON stage 20, comes out of a mirror. The high reflecting layer 20 and the piece of a prism form are a wrap about all cross sections (although shown to drawing 2 by 14, not shown in drawing 4 ), even if there are few reversible adjustable permeability fields of the reversible adjustable permeability device of an amelioration mirror.

[0098] The layer of the charge of a laminated wood does not exist in the adjustable reflection factor mirror (similar to what was shown in drawing 4 ) by which this invention of the form of further others was improved, but the electrode possession wall 100 is replaced by the piece of the charge of a prism profile, the surface of one of these is covered with a conductive layer, and this acts as an electrode of a reversible adjustable permeability device.

[0099] the amelioration mirror of this invention -- setting -- the angle of the plane of a reflective means or a reflecting layer (for example, drawing 3 layer 18A and drawing 4 layer 20), and a front face (for example, drawing 3 the surface 101 and drawing 4 surface 221) -- desirable -- about 1-- it is about 5 degrees.

[0100] The charge of a laminated wood of the layer 19 of the mirror 300 of drawing 3 and the layer 21 of the mirror 400 of drawing 4 may be what kind of transparent charge of a laminated wood known for this technical field. It is still better by any laminated layers methods by which the method of carrying out a laminating to the surface 101 is learned [ piece / the surface 131 or / 22 / prism form solid-state ] for this technical field in the prism form mirror 180 of a mirror 300. In the case of the desirable amelioration mirror (for example, mirror 300) of this invention which features for a reflective means to be a reflective means of a prism form mirror, the surface 131 is a glass piece, the solid material element 18 of a prism form mirror will be glass, and the transparent charge of a laminated wood is a polyvinyl butyral (PVB). Moreover, a reflective means adheres to one surface of a reversible adjustable permeability device directly. And the solid material piece of a prism form is the surface (this is parallel to the surface to which the reflective means has adhered) of a reversible adjustable

permeability device. this and a gap are kept -- \*\*\*\* -- it is a glass piece, the material piece of a prism form will be glass, and the case of the desirable amelioration mirror (for example, mirror 400) of this invention which features to carry out the laminating of the transparent charge of a laminated wood is [ a surface element 101 ] also PVB.

[0101] in drawing 3 and drawing 4 , a wall 100, its element 10 and the 10A; wall 130, its element 13, 13A; spacer 11; solution space 12; and a line, or a strip 16 is equivalent to the element of the same number of the device 200 shown in drawing 2 . The line or strip of drawing 3 and drawing 4 is extended even to the lead wire or the production corresponding to lead-wire 15A shown in drawing 2 .

[0102] The mirror of this invention is usually fixed to a frame, and this covers all the reversible adjustable permeability devices from a visual field except for most cross sections (shown to the device 200 of drawing 2 by 14) of the reversible adjustable permeability field through which it passes before and after it reflects with the reflective means of a mirror and a visible ray reflects in those who look at a mirror from a reflective means. The sense of a frame can be adjusted to hand control or an automatic target. The lead wire 15A and 16A (shown in drawing 2 ) of a device will be connected to the electric power supply controlling element (for example, a switching means, a means to control the inter-electrode potential difference). It could be located the device within the frame structure, and behind the reflective means by request, or dissociated completely with a frame and mounting, and these may connect these elements to a power supply (for example, cell). Although this kind of power supply (in the case of an especially small cell) may also be located in the frame structure, probably, the power supply (for example, cell for automobiles) is usually located in the outside of a frame. The use with the desirable adjustable reflection factor mirror of this invention is a thing as a glare prevention mold reflector glass for automobiles.

[0103] The framework of the device of this invention will essentially be carried out like the windowpane of an ordinary aperture or a windshield, when using as an adjustable permeability component part of an adjustable permeability color filter (especially aperture). Except for the portion corresponding to most cross sections (shown to the device 200 of drawing 2 by 14) of a reversible adjustable permeability field, all devices will be hidden from the visual field with the window frame component part. Similarly, the line from the lead wire 15A and 16A (shown in drawing 2 ) of a device will run the inside of this frame part article (outside of the visual field of an aperture), and will attain it to the external electric power supply means and external electric power supply controlling element of aperture structure.

[0104] A display device can be manufactured using one side or the both sides of the adjustable reflection factor mirror of this invention, and an adjustable reflection factor color filter. In this case, the device of this invention is a reversible adjustable permeability component part, and information is transmitted by change of a reflection factor or permeability. The field which luminous intensity is changed in the device of this invention, and is penetrated or reflected can be formed so that a display device may have the configuration of the mark made into the purpose. Or the separate this invention device can be arranged to suitable arrangement, and the configuration of the mark made into the purpose can also be given. In one gestalt, since a dark-colored mark is formed on the bright background of a device in connection with the permeability of a device (1 or 2 or more) falling, it will be in the condition of being visible to those whom the mark displayed looks at. In other gestalten, since a mark is in a dark background, when the mark is in sight in the state of the high permeability of a device, if a device (1 or 2 or more) is activated, permeability will fall and the mark will disappear from the visual field. With the display device using the device of this invention as a reversible adjustable permeability component part, almost all marks (an alphabetic character, a mark, a phrase, a number, or various kinds of designs are included) can be displayed. The display device using the adjustable permeability device of this invention is useful also in the aperture of the stained glass using a windowpane with a display of art, for example, reversible adjustable color.

[0105] This invention is explained more to details into the following examples.

[0106] Especially, it points, as long as there is nothing, all the concentration in an example can be set to a room temperature (20-27 degrees C), and all temperature is based on \*\*.

[0107] Example The cel was formed with the glass sheet (area 7.6cmx12.7cm) of 12 sheets, and it dissociated by the strip of a nylon monofilament with a thickness of 0.020cm. One side of these glass sheets is covered with the transparent electrode of the tin oxide (ITO) which carried out the indium dope, and has been arranged at the condition that these fields face mutually by the inside of a cel. As shown in drawing 2 , these sheets are shifted slightly mutually and gave the overhang strip of two parallel narrow ITO coats along the 12.7cm side (the capacity and the opposite side for solutions) of each sheet. By pasting up a copper strip with a conductive silver epoxy resin along with an overhang strip with this narrow ITO coat, the contact was formed and, subsequently the seal of the edge of a cel was carried out with the insulating epoxy resin. In front of the last seal, it was filled up with the propylene carbonate solution of N, N, N', N' - tetramethyl-1 and 4-phenylenediamines 0.05M, 1, and 1'-diheptyl -4, 4'-bipyridinium fluoroborate 0.05M, and tetrapod-n-butyl ammonium fluoroborate 0.5M in inter-electrode space.

[0108] When 1.0V were applied to inter-electrode, the solution which was colorless at first changed to the deep purple-blue color. When a cel was made into an open circuit condition, or when a cel was short-circuited, the solution returned to the decolorized colorless condition. When a cel was short-circuited after reversing the polarity of 1.0V inter-electrode potential for several seconds, the cel returned to the decolorization condition more promptly.

[0109] When silver plating of the surface (opposite side of an ITO covering surface) of one glass sheet was carried out, and seen through the glass of the side which has not carried out silver plating, the device had become an adjustable reflection factor mirror.

[0110] Example The cel which operates the glass sheet (area 10.2cmx10.2cm) of 22 sheets as an adjustable reflection factor mirror by opening a gap with a glass bead with a thickness of 0.013cm was created. One side of one glass sheet was covered with ITO, and one side of the glass sheet of another side was covered with the vacuum deposition layer of metal Inconel. ITO and each electrode layer of Inconel assembled the cel in the condition of facing mutually by the inside of a cel. The seal and array of the copper strip contact to an electrode surface and a device were the same as the case of the cel of Example 1. inter-electrode space -- 5, 10-dihydro-5, 10-dimethyl phenazine 0.02M, and tetramethylen-screw [4-(1-benzyl pyridine-4'-IRU) pyridinium] tetrafluoroborate 0.02M -- and -- tetra--- it was filled up with the solution in the propylene carbonate of -n-butyl ammonium fluoroborate 0.1M.

[0111] When the potential of 1.0V was given to ITO and Inconel inter-electrode, the reflection factor from the Inconel electrode fell quickly. The solution layer changed with the given potentials to the deep bluish green color. When the given potential was removed, the solution returned to bright zero potential equilibrium, and the reflection factor from the Inconel electrode increased even to the high level of the beginning before giving the potential difference.

[0112] Example By detaching 0.013cm and arranging using a glass bead, by carrying out the glass sheet of two sheets covered with ITO in 3 one side to space formation, the device which operates as an adjustable permeability color filter or an aperture was created. The size of a glass sheet was 6.4cmx25.4cm. The ITO covering surface of each sheet was opposed. The sheet and array of a copper strip contact and a device are the same as the case of the device of Example 1, and have arranged the strip along with the 25.4cm side of a seal. They are 1 and 1'-dibenzyl to inter-electrode space. - 4, 4'-JIPRIJINIUMU difluoro borate 0.05M and 5, 10-dihydro - It was filled up with the solution in 5 and the propylene carbonate of 10-dimethyl phenazine 0.05M.

[0113] When the voltage of 1.1V was given to the inter-electrode solution layer, the white light permeability of a device fell to 10.0% from 81.5% within 11 seconds. The steady state permeability of the device at the time of giving 1.1V was 6.0%. When the electrode was short-circuited, the permeability of a device increased from 10% to 70% within 7 seconds, and permeability returned to 81.5% within 16 seconds after the electrode short circuit. It was made to circulate 40,000 times between the steady states which gave permeability [ in / for a device / the zero potential ], and the potential of 1.1V to inter-electrode at the room temperature. After 40,000 times, the permeability of the device in zero potential is 78.5%, and the steady state permeability at the time of giving the potential of 1.1V was only 6.0%. The speed of change of permeability did not change with circulation.

[0114] Although the permeability in zero potential fell to 71.5% when circulating a device further 20,000 times in 55 degrees C between zero potential and the steady state permeability of 1.1V, the permeability in 1.1V was only 6.0%.

[0115] example the device of 4 example 3 -- the same -- carrying out -- however, inter-electrode space -- 1 and 1' - G (n-heptyl) -4 and 4' - bipyridinium difluoro borate 0.04M and 5, 10-dihydro-5, and 10-dimethyl phenazine 0.04M -- and -- tetra--- the device which operates as an adjustable permeability color filter was created by being filled up with the solution in the propylene carbonate of -n-butyl ammonium fluoroborate 0.1M.

[0116] The white light permeability of a device fell to 20.0% from 84.5% within 10 seconds by giving the potential of 1.1V to an inter-electrode solution layer. The device steady state permeability at the time of giving 1.1V was 11.0%. The permeability of the device at the time of short-circuiting an electrode increased from 20% to 70% within 7 seconds, and after short-circuiting an electrode, permeability returned to 84.5% within 22 seconds. The device was circulated 40,000 times between the steady state permeability at the time of zero potential permeability and 1.1V being given by inter-electrode at a room temperature. The permeability in the condition of zero potential permeability being 84.0% and having given the potential of 1.1V after 40,000 times was 11.0%. The speed of change of permeability did not change with circulation.

[0117] When circulating a device further 20,000 times between zero potential permeability and the steady state permeability in 1.1V at 55 degrees C, the permeability in zero potential fell to 77.5%, and the permeability in 1.1V was only 11.0%.

[0118] Example Five devices which set the size of an ITO covering glass sheet to 6.4cmx7.6cm, however operate as an adjustable permeability color filter or an aperture as well as the device of 3 were created. An inter-electrode solution is 1 in propylene carbonate, the 1'-dibenzyl -4, 4'-bipyridinium difluoro borate 0.05M and 5, and 10-dihydro. - It was 5 and 10-dimethyl phenazine 0.05M.

[0119] When the potential of 1.1V was given to the inter-electrode solution, permeability fell [ the white light of a device ] to 10.0% from 81.5% within 10 seconds. The steady state permeability of the device at the time of giving 1.1V was 11.0%. The permeability of the device at the time of short-circuiting an electrode increased from 20% to 70% within 6 seconds, and after short-circuiting an electrode, permeability returned to 81.5% within 15 seconds. It was made to circulate 40,000 times between the permeability [ in / for a device / at 55 degrees C / zero potential ], and its steady state permeability at the time of giving 1.1V to inter-electrode. After 40,000 times, zero potential permeability is 65.0% and the steady state permeability in the potential of 1.1V was only 6.0%. The speed of change of permeability did not change by this circulation.

[0120] Example Six devices which operate as an adjustable permeability color filter were created like 3. However, it is N, N, N, and N'-tetramethyl about inter-electrode space. - 1 4-phenylenediamine 0.01M, 5, 10-dihydro - 5, 10-dimethyl phenazines 0.01M and 1, the 1'-dibenzyl -4, 4'-bipyridinium difluoro borate 0.01M, tetramethylen-screw-[4-(1-benzyl pyridine-4'-IRU) pyridinium] tetrafluoroborate 0.01M -- and -- tetra--- it was filled up with the solution in the propylene carbonate of -n-butyl ammonium fluoroborate 0.1M.

[0121] When the potential of 1.2V was given to the inter-electrode solution, the white light permeability of a device fell to 10% from 84% within 4 seconds. The steady state permeability in 1.2V was 5%. When the electrode was short-circuited, the permeability of a device increased from 10% to 70% within 6.5 seconds, and after it short-circuited the electrode, it returned to 84% of zero potential balanced values of a basis within 15 seconds.

[0122] Example It created by the same method as the device and the essential target which showed seven examples to 3, and it was admitted that the device filled up with the propylene carbonate solution of the electrochromic compound shown in the following table 1 operated as a self-elimination type solution phase electrochromic device like what was shown in Examples 1-6.

[0123]

[A table 1]

表 1

	アノードエレクト ロクロミック化合物	カソードエレクトロクロ ミック化合物
1.	5,10-ジヒドロ-5, 10-ジメチルフエナ ジン	1,1'-ジ- ( n -ヘプチル ) - 4,4'-ビビリジニウムジフ ルオロボレート
2.	5,10-ジヒドロ-5, 10-ジメチルフエナ ジン	1,1'-ジ- ( n -ヘプチル ) - 4,4'-ビビリジニウムジバ ークロレート
3.	5,10-ジヒドロ-5, 10-ジメチルフエナ ジン	1,1'-ジフェニル-4,4'-ビ ビリジニウムジフルオロボレ ート
4.	10-メチルフエノチ アジン	1,1'-ジ- ( n -ヘプチル ) - 4,4'-ビビリジニウムジフ ルオロボレート
5.	10-エチルフエノチ アジン	1,1'-ジ- ( n -ヘプチル ) - 4,4'-ビビリジニウムジフ ルオロボレート
6.	テトラテアフルバレン	1,1'-ジベンジル-4,4'-ビ ビリジニウムジフルオロボレー ト

[0124] Example The compound of 8 a large number was examined per [ as an anode or a cathode electrochromic compound ] acceptance possibility by using propylene carbonate as a solvent in the single partition mold self-elimination type solution phase device of this invention.

[0125] It was admitted that it could not receive on the occasion of a certain kind of compound \*\*\*\*\* (cathode compound) or oxidation (anode compound) since it is unstable. This instability is chemically shown by a reversible reduction wave (in the case of a cathode compound), or when a reversible oxidation wave (in the case of an anode compound) does not exist at all chemically or only one piece exists in Volta MOGURAFU which has set to the room temperature in the solvent per that compound, shifted, and was obtained by that standard method.

[0126] In a solvent the reversible Volta MOGURAFU reduction wave (in the case of a cathode compound), or at

least two compounds which have the reversible Volta MOGURAFU oxidation wave (in the case of an anode compound) chemically on at least two chemistry targets in the above-mentioned solvent Having not resulted in instability (especially circulation being received) to the extent that it cannot receive, even if it combines with one sort of what kind of compounds of others with the same property or two sorts or more was admitted.

[0127] In order to be what can be received, an absorbancy index must change [ in / on the occasion of reduction or oxidation in a solvent / in a compound / at least one sort of wavelength of a visible region (4200-700A) ] clearly. In order to guarantee stability, change of this kind takes place with at least two sorts of reduction chemically corresponding to the 1st thing among reversible BORUTAMO graph reduction waves, when a compound is a cathode compound, and when a compound is an anode compound, it must take place with at least two sorts of oxidation chemically corresponding to a reversible BORUTAMO graph oxidation wave.

[0128] Can carry out minimum acceptance as a cathode or an anode electrochromic compound into the solvent of this invention, and also Have the solubility of  $10^{-4}$ M and it sets on at least one sort of wavelength of a visible region. a compound -- desirable -- the inside of the solvent of the solution -- the zero potential equilibrium -- setting -- at least -- about  $10^{-2}$  -- In the case of a cathode compound, the 1st reduction chemically corresponding to the reversible Volta MOGURAFU reduction wave is faced. or the case of an anode compound -- the 1st oxidation chemically corresponding to the reversible Volta MOGURAFU oxidation wave -- facing -- an absorbancy index -- at least -- about  $10^{-2}$  -- only the factor of  $10^3 \text{cm}^{-1} \text{M}^{-1}$  increases at least -- I will come out.

[0129] The compound with which having suited the criteria of these acceptance possibilities and desirability by using propylene carbonate as a solvent was admitted altogether The thing explained in full detail to either of Examples 1-7 and the still newer anode compound N, N', N''-trimethyl TORIFENA gin oxazine, Known anode compound ortho toluidine, N and N, N', an N'-tetramethyl benzidine, N, N, N', and N' -- the - tetra-phenyl -1 and 4-phenylenediamine -- And 5, 10-dihydro - 5, 10-diphenyl phenazine and the known cathode compound 1, the 1'-dimethyl -4, 4'-bipyridinium dichloride, They are 1, 1'-G (p-cyanophenyl) -4, 4'-bipyridinium difluoro borate and 1, 1'-diphenyl -4, and 4'-bipyridinium JIYOJIDO.

[0130] example 9 -- this example is adjusted to a mean value with the darkest value that can be attained without spoiling a permeability "bright (clear)" (namely, zero potential balance) value and chemical stability when the device of this invention adjusts a gray-scale device, i.e., the inter-electrode potential difference, and it is shown that it is useful as a device which can be stabilized here.

[0131] The same cel as the thing of Example 3 is constituted, and they are 1 in propylene carbonate, and 1'-G (n-heptyl). - 4, 4'-bipyridinium difluoro borate 0.04M and 5, 10-dihydro - It was filled up with the solution of 5 and 10-diphenyl phenazine 0.04M. The steady state permeability to the white light was measured as a function of the inter-electrode potential difference of a device, and the value shown in a table 2 was acquired.

[0132]

[A table 2]

表 2

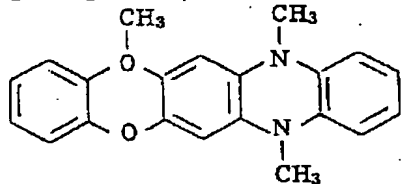
電極間の電位 (V)	定常状態透過率 (%)
0.0	8.3
0.1	8.3
0.2	8.3
0.3	8.3
0.4	8.1.5
0.5	7.1.5
0.6	5.6.0
0.7	4.2.0
0.8	3.1.0
0.9	2.4.0
1.0	1.7.0
1.1	1.3.0
1.2	1.1.5



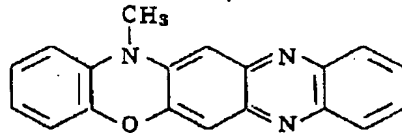
[0133] Example It created by applying the dichloroethane solution of acrylic sheet material Lucite L to the ITO surface of the glass piece which carried out ITO covering of the device which operates as an adjustable reflection factor mirror containing the solution thickened ten times. When dichloroethane was evaporated, the thin film (weight of 0.29g) of an acrylic sheet material was left behind to the ITO surface. this same glass piece equips an ITO-acrylic material side and the opposite side with the reflexivity mirror, i.e., a silver coat, and created the cel by separating that ITO-acrylic side from the ITO side of the 2nd person glass piece (one field is equipped only with the ITO coat) 0.013cm, and arranging it. The size of a glass sheet was 6.4cmx25.4cm. The structure of a copper strip contact, a seal, and a device was the same as the case of Example 3. They are 1 and 1'-G n-heptyl to the space between the ITO sides of one glass piece. - 4, 4'-bipyridinium difluoro borate 0.04M and 5, 10-dihydro - It was filled up with the solution in the propylene carbonate of 5, 10-diphenyl phenazine 0.04M, and tetrabutylammonium fluoroborate 0.1M.

[0134] At the room temperature, it was able to dissolve in the propylene carbonate solution, the acrylic layer was able to be thickened within several hours, and this device was able to be operated as an adjustable reflection factor mirror by changing the potential given to the solution between ITO electrode layers. Where the voltage of 1.2V is impressed, the reflection factor changed to 20.0% from 73.5% within 2.5 seconds, and the steady state reflection factor of \*\* was reached 9.0%. When the electrode was short-circuited, the reflection factor increased from 9.0% to 60.0% within 17 seconds, and, finally returned to 73.5% of bright zero potential values of a basis.

[0135] Example 11 Ns, N', composition of N''-trimethyl TORIFENA gin oxazine [\*\* 47]



The known compound of a degree type, N-MECHIRUTORI phenazine oxazine [\*\* 48]



since -- it left, and in order to change phenazine into 5, 10-dihydro-5, and 10-dimethyl phenazine, said compound was manufactured according to the method indicated by Guillemin and Diedrich at the journal OBU American chemical societies 79 and 6178 (1957). 100mg [ of the above-mentioned starting material ] (0.33 millimol), 25mg [ of metallic potassium ] (0.67 millimol), and ethylene glycol wood ether 5ml was agitated for 12 hours. Subsequently, absolute alcohol was added and the superfluous potassium was disassembled, after adding a superfluous methyl iodide.

[0136] Subsequently, the reaction mixture was mixed with water. The produced precipitate was \*\*\*\*\*ed from ethanol and about 2mg of pure products was obtained.

[0137] This product is a reversible oxidation wave and 5, and 10-dihydro chemically in propylene carbonate. - It was admitted that the case of 5 and 10-dimethyl phenazine and change of an extremely similar color were shown.

[0138] Example By carrying out the laminating of the same electrochromic device as the thing of Example 3 to the common reflector glass for prism form automobiles using the transparent charge polyvinyl butyral (PVB) of a laminated wood by the 12 standard method, the device of the structure shown in drawing 3 was created. They are 1 and 1'-dibenzyl to this device. - 4, 4'-bipyridinium difluoro borate 0.02M and 5, 10-dihydro - It was filled up with the solution in the propylene carbonate of 5, 10-dimethyl phenazine 0.02M, and tetrapod-n-butyl ammonium fluoroborate 0.1M. This device was used as a reflector glass in an automobile. This device gave the continuation adjustable reflection factor (namely, gray scale) mirror without distortion during actuation. This was very effective in removing the glare by the headlight of the automobile which approaches from back during Nighttime operation.

[0139] The glare from the headlight of the automobile which this device approaches from back was a zero potential difference, most or when there was completely nothing, and in the case of the glare of whenever [ middle ], it is 0.6V, and, in the case of the high glare, operated by 1.0V.

[0140] In zero potential, the \*\*\*\*\* reflection factor from the silver plating side of a prism mirror was 70% or more of a light which carries out incidence to a device. The potential of 1.0V to which, as for the steady state reflection factor from the silver plating surface, the given potential was given about 30% by 0.6V was about 10% of reflection factors.



[0141] As mentioned above, although this invention was described to some extent in details, these various modification and corrections will be recognized by this contractor within the limits of the pneuma of this invention. These modification and corrections are also indicated by this specification, and are included in the range of this invention by which the patent claim was carried out.

---

[Translation done.]

\* NOTICES \*

Japan Patent Office is not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

---

MEANS

---

It will connect with for example, (a switching means and a means to control the inter-electrode potential difference). It could be located the device within the frame structure, and behind the reflective means by request, or dissociated completely with a frame and mounting, and these may connect these elements to a power supply (for example, cell). Although this kind of power supply (in the case of an especially small cell) may also be located in the frame structure, probably, the power supply (for example, cell for automobiles) is usually located in the outside of a frame. The use with the desirable adjustable reflection factor mirror of this invention is a thing as a glare prevention mold reflector glass for automobiles.

[0103] The framework of the device of this invention will essentially be carried out like the windowpane of an ordinary aperture or a windshield, when using as an adjustable permeability component part of an adjustable permeability color filter (especially aperture). Except for the portion corresponding to most cross sections (shown to the device 200 of drawing 2 by 14) of a reversible adjustable permeability field, all devices will be hidden from the visual field with the window frame component part. Similarly, the line from the lead wire 15A and 16A (shown in drawing 2) of a device will run the inside of this frame part article (outside of the visual field of an aperture), and will attain it to the external electric power supply means and external electric power supply controlling element of aperture structure.

[0104] A display device can be manufactured using one side or the both sides of the adjustable reflection factor mirror of this invention, and an adjustable reflection factor color filter. In this case, the device of this invention is a reversible adjustable permeability component part, and information is transmitted by change of a reflection factor or permeability. The field which luminous intensity is changed in the device of this invention, and is penetrated or reflected can be formed so that a display device may have the configuration of the mark made into the purpose. Or the separate this invention device can be arranged to suitable arrangement, and the configuration of the mark made into the purpose can also be given. In one gestalt, since a dark-colored mark is formed on the bright background of a device in connection with the permeability of a device (1 or 2 or more) falling, it will be in the condition of being visible to those whom the mark displayed looks at. In other gestalten, since a mark is in a dark background, when the mark is in sight in the state of the high permeability of a device, if a device (1 or 2 or more) is activated, permeability will fall and the mark will disappear from the visual field. With the display device using the device of this invention as a reversible adjustable permeability component part, almost all marks (an alphabetic character, a mark, a phrase, a number, or various kinds of designs are included) can be displayed. The display device using the adjustable permeability device of this invention is useful also in the aperture of the stained glass using a windowpane with a display of art, for example, reversible adjustable color.

[0105] This invention is explained more to details into the following examples.

[0106] Especially, it points, as long as there is nothing, all the concentration in an example can be set to a room temperature (20-27 degrees C), and all temperature is based on \*\*.

[0107] Example The cel was formed with the glass sheet (area 7.6cmx12.7cm) of 12 sheets, and it dissociated by the strip of a nylon monofilament with a thickness of 0.020cm. One side of these glass sheets is covered with the transparent electrode of the tin oxide (ITO) which carried out the indium dope, and has been arranged at the condition that these fields face mutually by the inside of a cel. As shown in drawing 2, these sheets are shifted slightly mutually and gave the overhang strip of two parallel narrow ITO coats along the 12.7cm side (the capacity and the opposite side for solutions) of each sheet. By pasting up a copper strip with a conductive silver epoxy resin along with an overhang strip with this narrow ITO coat, the contact was formed and, subsequently the seal of the edge of a cel was carried out with the insulating epoxy resin. In front of the last seal, it was filled up with the propylene carbonate solution of N, N, N', N' - tetramethyl-1 and 4-phenylenediamines 0.05M, 1, and 1'-diheptyl -4, 4'-bipyridinium fluoroborate 0.05M, and tetrapod-n-butyl ammonium fluoroborate 0.5M in inter-electrode space.

[0108] When 1.0V were applied to inter-electrode, the solution which was colorlessness at first changed to the deep purple-blue color. When a cel was made into an open circuit condition, or when a cel was short-circuited, the solution returned to the decolorized colorless condition. When a cel was short-circuited after reversing the

polarity of 1.0V inter-electrode potential for several seconds, the cel returned to the decolorization condition more promptly.

[0109] When silver plating of the surface (opposite side of an ITO covering surface) of one glass sheet was carried out, and seen through the glass of the side which has not carried out silver plating, the device had become an adjustable reflection factor mirror.

[0110] Example The cel which operates the glass sheet (area 10.2cmx10.2cm) of 22 sheets as an adjustable reflection factor mirror by opening a gap with a glass bead with a thickness of 0.013cm was created. One side of one glass sheet was covered with ITO, and one side of the glass sheet of another side was covered with the vacuum deposition layer of metal Inconel. ITO and each electrode layer of Inconel assembled the cel in the condition of facing mutually by the inside of a cel. The seal and array of the copper strip contact to an electrode surface and a device were the same as the case of the cel of Example 1. inter-electrode space -- 5, 10-dihydro-5, 10-dimethyl phenazine 0.02M, and tetramethylen-screw [4-(1-benzyl pyridine-4'-IRU) pyridinium] tetrafluoroborate 0.02M -- and -- tetra--- it was filled up with the solution in the propylene carbonate of -n-butyl ammonium fluoroborate 0.1M.

[0111] When the potential of 1.0V was given to ITO and Inconel inter-electrode, the reflection factor from the Inconel electrode fell quickly. The solution layer changed with the given potentials to the deep bluish green color. When the given potential was removed, the solution returned to bright zero potential equilibrium, and the reflection factor from the Inconel electrode increased even to the high level of the beginning before giving the potential difference.

[0112] Example By detaching 0.013cm and arranging using a glass bead, by carrying out the glass sheet of two sheets covered with ITO in 3 one side to space formation, the device which operates as an adjustable permeability color filter or an aperture was created. The size of a glass sheet was 6.4cmx25.4cm. The ITO covering surface of each sheet was opposed. The sheet and array of a copper strip contact and a device are the same as the case of the device of Example 1, and have arranged the strip along with the 25.4cm side of a seal. They are 1 and 1'-dibenzyl to inter-electrode space. - 4, 4'-JIPRIJINIUMU difluoro borate 0.05M and 5, 10-dihydro - It was filled up with the solution in 5 and the propylene carbonate of 10-dimethyl phenazine 0.05M.

[0113] When the voltage of 1.1V was given to the inter-electrode solution layer, the white light permeability of a device fell to 10.0% from 81.5% within 11 seconds. The steady state permeability of the device at the time of giving 1.1V was 6.0%. When the electrode was short-circuited, the permeability of a device increased from 10% to 70% within 7 seconds, and permeability returned to 81.5% within 16 seconds after the electrode short circuit. It was made to circulate 40,000 times between the steady states which gave permeability [ in / for a device / the zero potential ], and the potential of 1.1V to inter-electrode at the room temperature. After 40,000 times, the permeability of the device in zero potential is 78.5%, and the steady state permeability at the time of giving the potential of 1.1V was only 6.0%. The speed of change of permeability did not change with circulation.

[0114] Although the permeability in zero potential fell to 71.5% when circulating a device further 20,000 times in 55 degrees C between zero potential and the steady state permeability of 1.1V, the permeability in 1.1V was only 6.0%.

[0115] example the device of 4 example 3 -- the same -- carrying out -- however, inter-electrode space -- 1 and 1' - G (n-heptyl) -4 and 4' - bipyridinium difluoro borate 0.04M and 5, 10-dihydro-5, and 10-dimethyl phenazine 0.04M -- and -- tetra--- the device which operates as an adjustable permeability color filter was created by being filled up with the solution in the propylene carbonate of -n-butyl ammonium fluoroborate 0.1M.

[0116] The white light permeability of a device fell to 20.0% from 84.5% within 10 seconds by giving the potential of 1.1V to an inter-electrode solution layer. The device steady state permeability at the time of giving 1.1V was 11.0%. The permeability of the device at the time of short-circuiting an electrode increased from 20% to 70% within 7 seconds, and after short-circuiting an electrode, permeability returned to 84.5% within 22 seconds. The device was circulated 40,000 times between the steady state permeability at the time of zero potential permeability and 1.1V being given by inter-electrode at a room temperature. The permeability in the condition of zero potential permeability being 84.0% and having given the potential of 1.1V after 40,000 times was 11.0%. The speed of change of permeability did not change with circulation.

[0117] When circulating a device further 20,000 times between zero potential permeability and the steady state permeability in 1.1V at 55 degrees C, the permeability in zero potential fell to 77.5%, and the permeability in 1.1V was only 11.0%.

[0118] Example Five devices which set the size of an ITO covering glass sheet to 6.4cmx7.6cm, however operate as an adjustable permeability color filter or an aperture as well as the device of 3 were created. An inter-electrode solution is 1 in propylene carbonate, the 1'-dibenzyl -4, 4'-bipyridinium difluoro borate 0.05M and 5, and 10-dihydro. - It was 5 and 10-dimethyl phenazine 0.05M.

[0119] When the potential of 1.1V was given to the inter-electrode solution, permeability fell [ the white light of a device ] to 10.0% from 81.5% within 10 seconds. The steady state permeability of the device at the time of giving 1.1V was 11.0%. The permeability of the device at the time of short-circuiting an electrode increased from

20% to 70% within 6 seconds, and after short-circuiting an electrode, permeability returned to 81.5% within 15 seconds. It was made to circulate 40,000 times between the permeability [ in / for a device / at 55 degrees C / zero potential ], and its steady state permeability at the time of giving 1.1V to inter-electrode. After 40,000 times, zero potential permeability is 65.0% and the steady state permeability in the potential of 1.1V was only 6.0%. The speed of change of permeability did not change by this circulation.

[0120] Example Six devices which operate as an adjustable permeability color filter were created like 3. However, it is N, N, N, and N'-tetramethyl about inter-electrode space. - 1 4-phenylenediamine 0.01M, 5, 10-dihydro - 5, 10-dimethyl phenazines 0.01M and 1, the 1'-dibenzyl -4, 4'-bipyridinium difluoro borate 0.01M, tetramethylen-screw-[4-(1-benzyl pyridine-4'-IRU) pyridinium] tetrafluoroborate 0.01M -- and -- tetra--- it was filled up with the solution in the propylene carbonate of -n-butyl ammonium fluoroborate 0.1M.

[0121] When the potential of 1.2V was given to the inter-electrode solution, the white light permeability of a device fell to 10% from 84% within 4 seconds. The steady state permeability in 1.2V was 5%. When the electrode was short-circuited, the permeability of a device increased from 10% to 70% within 6.5 seconds, and after it short-circuited the electrode, it returned to 84% of zero potential balanced values of a basis within 15 seconds.

[0122] Example It created by the same method as the device and the essential target which showed seven examples to 3, and it was admitted that the device filled up with the propylene carbonate solution of the electrochromic compound shown in the following table 1 operated as a self-elimination type solution phase electrochromic device like what was shown in Examples 1-6.

[0123]

[A table 1]

表 1

	アノードエレクト ロクロミック化合物	カソードエレクトロクロ ミック化合物
1.	5,10-ジヒドロ-5, 10-ジメチルフエナ ジン	1,1'-ジ- ( n -ヘプチル ) - 4,4'-ビビリジニウムジフ ルオロボレート
2.	5,10-ジヒドロ-5, 10-ジメチルフエナ ジン	1,1'-ジ- ( n -ヘプチル ) - 4,4'-ビビリジニウムジバ ークロレート
3.	5,10-ジヒドロ-5, 10-ジメチルフエナ ジン	1,1'-ジフェニル-4,4'-ビ ビリジニウムジフルオロボレ ート
4.	10-メチルフエノチ アジン	1,1'-ジ- ( n -ヘプチル ) - 4,4'-ビビリジニウムジフ ルオロボレート
5.	10-エチルフエノチ アジン	1,1'-ジ- ( n -ヘプチル ) - 4,4'-ビビリジニウムジフ ルオロボレート
6.	テトラテアフルバレン	1,1'-ジベンジル-4,4'-ビ ビリジニウムジフルオロボレ ート

[0124] Example The compound of 8 a large number was examined per [ as an anode or a cathode electrochromic compound ] acceptance possibility by using propylene carbonate as a solvent in the single partition mold self-elimination type solution phase device of this invention.

[0125] It was admitted that it could not receive on the occasion of a certain kind of compound \*\*\*\*\* (cathode compound) or oxidation (anode compound) since it is unstable. This instability is chemically shown by a reversible reduction wave (in the case of a cathode compound), or when a reversible oxidation wave (in the case of an anode compound) does not exist at all chemically or only one piece exists in Volta MOGURAFU which has set to the room temperature in the solvent per that compound, shifted, and was obtained by that standard method.

[0126] In a solvent the reversible Volta MOGURAFU reduction wave (in the case of a cathode compound), or at least two compounds which have the reversible Volta MOGURAFU oxidation wave (in the case of an anode compound) chemically on at least two chemistry targets in the above-mentioned solvent Having not resulted in instability (especially circulation being received) to the extent that it cannot receive, even if it combines with

one sort of what kind of compounds of others with the same property or two sorts or more was admitted.

[0127] In order to be what can be received, an absorbancy index must change [ in / on the occasion of reduction or oxidation in a solvent / in a compound / at least one sort of wavelength of a visible region (4200-700A) ] clearly. In order to guarantee stability, change of this kind takes place with at least two sorts of reduction chemically corresponding to the 1st thing among reversible BORUTAMO graph reduction waves, when a compound is a cathode compound, and when a compound is an anode compound, it must take place with at least two sorts of oxidation chemically corresponding to a reversible BORUTAMO graph oxidation wave.

[0128] Can carry out minimum acceptance as a cathode or an anode electrochromic compound into the solvent of this invention, and also Have the solubility of -4M and it sets on at least one sort of wavelength of a visible region. a compound -- desirable -- the inside of the solvent of the solution -- the zero potential equilibrium -- setting -- at least -- about 10 -- In the case of a cathode compound, the 1st reduction chemically corresponding to the reversible Volta MOGURAFU reduction wave is faced. or the case of an anode compound -- the 1st oxidation chemically corresponding to the reversible Volta MOGURAFU oxidation wave -- facing -- an absorbancy index -- at least -- about  $10^{-2}$  -- only the factor of  $10^3\text{cm}^{-1}\text{M}^{-1}$  increases at least -- I will come out.

[0129] The compound with which having suited the criteria of these acceptance possibilities and desirability by using propylene carbonate as a solvent was admitted altogether The thing explained in full detail to either of Examples 1-7 and the still newer anode compound N, N', N''-trimethyl TORIFENA gin oxazine, Known anode compound ortho toluidine, N and N, N', an N'-tetramethyl benzidine, N, N, N', and N' -- the - tetra-phenyl -1 and 4-phenylenediamine -- And 5, 10-dihydro - 5, 10-diphenyl phenazine and the known cathode compound 1, the 1'-dimethyl -4, 4'-bipyridinium dichloride, They are 1, 1'-G (p-cyanophenyl) -4, 4'-bipyridinium difluoro borate and 1, 1'-diphenyl -4, and 4'-bipyridinium JIYOJIDO.

[0130] example 9 -- this example is adjusted to a mean value with the darkest value that can be attained without spoiling a permeability "bright (clear)" (namely, zero potential balance) value and chemical stability when the device of this invention adjusts a gray-scale device, i.e., the inter-electrode potential difference, and it is shown that it is useful as a device which can be stabilized here.

[0131] The same cel as the thing of Example 3 is constituted, and they are 1 in propylene carbonate, and 1'-G (n-heptyl). - 4, 4'-bipyridinium difluoro borate 0.04M and 5, 10-dihydro - It was filled up with the solution of 5 and 10-diphenyl phenazine 0.04M. The steady state permeability to the white light was measured as a function of the inter-electrode potential difference of a device, and the value shown in a table 2 was acquired.

[0132]

[A table 2]

表 2

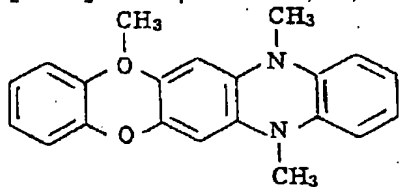
電極間の電位 (V)	定常状態透過率 (%)
0.0	8.3
0.1	8.3
0.2	8.3
0.3	8.3
0.4	8.1.5
0.5	7.1.5
0.6	5.6.0
0.7	4.2.0
0.8	3.1.0
0.9	2.4.0
1.0	1.7.0
1.1	1.3.0
1.2	1.1.5

[0133] Example It created by applying the dichloroethane solution of acrylic sheet material Lucite L to the ITO surface of the glass piece which carried out ITO covering of the device which operates as an adjustable

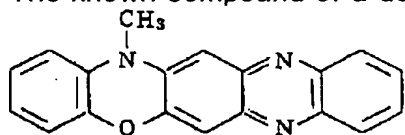
reflection factor mirror containing the solution thickened ten times. When dichloroethane was evaporated, the thin film (weight of 0.29g) of an acrylic sheet material was left behind to the ITO surface. this same glass piece equips an ITO-acrylic material side and the opposite side with the reflexivity mirror, i.e., a silver coat, and created the cel by separating that ITO-acrylic side from the ITO side of the 2nd person glass piece (one field is equipped only with the ITO coat) 0.013cm, and arranging it. The size of a glass sheet was 6.4cmx25.4cm. The structure of a copper strip contact, a seal, and a device was the same as the case of Example 3. They are 1 and 1'-G n-heptyl to the space between the ITO sides of one glass piece. - 4, 4'-bipyridinium difluoro borate 0.04M and 5, 10-dihydro - It was filled up with the solution in the propylene carbonate of 5, 10-diphenyl phenazine 0.04M, and tetrabutylammonium fluoroborate 0.1M.

[0134] At the room temperature, it was able to dissolve in the propylene carbonate solution, the acrylic layer was able to be thickened within several hours, and this device was able to be operated as an adjustable reflection factor mirror by changing the potential given to the solution between ITO electrode layers. Where the voltage of 1.2V is impressed, the reflection factor changed to 20.0% from 73.5% within 2.5 seconds, and the steady state reflection factor of \*\* was reached 9.0%. When the electrode was short-circuited, the reflection factor increased from 9.0% to 60.0% within 17 seconds, and, finally returned to 73.5% of bright zero potential values of a basis.

[0135] Example 11 Ns, N', composition of N''-trimethyl TORIFENA gin oxazine [\*\* 47]



The known compound of a degree type, N-MECHIRUTORI phenazine oxazine [\*\* 48]



since -- it left, and in order to change phenazine into 5, 10-dihydro-5, and 10-dimethyl phenazine, said compound was manufactured according to the method indicated by Guillemain and Diedrich at the journal OBU American chemical societies 79 and 6178 (1957). 100mg [ of the above-mentioned starting material ] (0.33 millimol), 25mg [ of metallic potassium ] (0.67 millimol), and ethylene glycol wood ether 5ml was agitated for 12 hours. Subsequently, absolute alcohol was added and the superfluous potassium was disassembled, after adding a superfluous methyl iodide.

[0136] Subsequently, the reaction mixture was mixed with water. The produced precipitate was \*\*\*\*\* from ethanol and about 2mg of pure products was obtained.

[0137] This product is a reversible oxidation wave and 5, and 10-dihydro chemically in propylene carbonate. - It was admitted that the case of 5 and 10-dimethyl phenazine and change of an extremely similar color were shown.

[0138] Example By carrying out the laminating of the same electrochromic device as the thing of Example 3 to the common reflector glass for prism form automobiles using the transparent charge polyvinyl butyral (PVB) of a laminated wood by the 12 standard method, the device of the structure shown in drawing 3 was created. They are 1 and 1'-dibenzyl to this device. - 4, 4'-bipyridinium difluoro borate 0.02M and 5, 10-dihydro - It was filled up with the solution in the propylene carbonate of 5, 10-dimethyl phenazine 0.02M, and tetrapod-n-butyl ammonium fluoroborate 0.1M. This device was used as a reflector glass in an automobile. This device gave the continuation adjustable reflection factor (namely, gray scale) mirror without distortion during actuation. This was very effective in removing the glare by the headlight of the automobile which approaches from back during Nighttime operation.

[0139] The glare from the headlight of the automobile which this device approaches from back was a zero potential difference, most or when there was completely nothing, and in the case of the glare of whenever [ middle ], it is 0.6V, and, in the case of the high glare, operated by 1.0V.

[0140] In zero potential, the \*\*\*\*\* reflection factor from the silver plating side of a prism mirror was 70% or more of a light which carries out incidence to a device. The potential of 1.0V to which, as for the steady state reflection factor from the silver plating surface, the given potential was given about 30% by 0.6V was about 10% of reflection factors.

[0141] As mentioned above, although this invention was described to some extent in details, these various modification and corrections will be recognized by this contractor within the limits of the pneuma of this invention. These modification and corrections are also indicated by this specification, and are included in the

range of this invention by which the patent claim was carried out.

---

[Translation done.]

**\* NOTICES \***

Japan Patent Office is not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

---

**DESCRIPTION OF DRAWINGS**

---

[Brief Description of the Drawings]

[Drawing 1] Drawing 1 shows the exploded view of the electrochromic device of this invention roughly.

[Drawing 2] Drawing 2 shows roughly the part assembly drawing of the electrochromic device of this invention.

[Drawing 3] Drawing 3 shows roughly the cross section of the adjustable reflection factor mirror of this invention assembled partially.

[Drawing 4] Drawing 4 shows roughly the cross section of the adjustable reflection factor mirror of this invention assembled partially.

[Description of Notations]

In each drawing, a mark expresses the following.

200: Electrochromic device

100,130: Electrode possession side plate

10 13: Solid material layer

10A, 13A: Electrode layer

11: Spacer

11A, 11B, 11C, the common-law marriage of 11D:11

12: Solution space

15 16: A conductive strip (line)

15A, 16A: Lead wire

18A, 20: Reflective means

300,400: Mirror

18 22: The piece of the charge of a prism profile

19 21: Laminating material layer

---

[Translation done.]



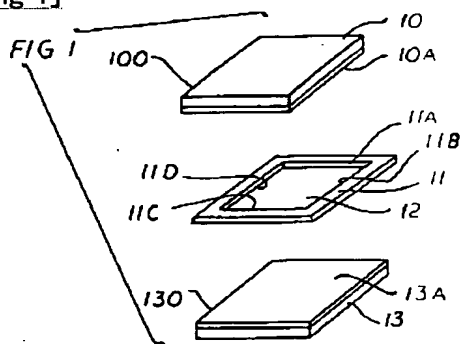
**\* NOTICES \***

Japan Patent Office is not responsible for any damages caused by the use of this translation.

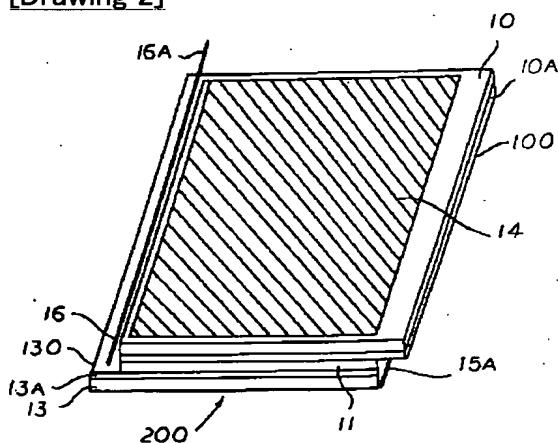
- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

**DRAWINGS**

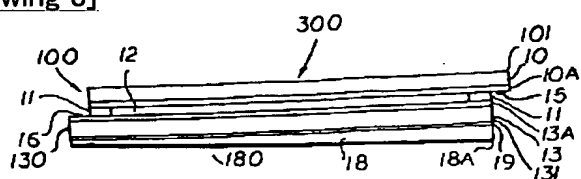
**[Drawing 1]**



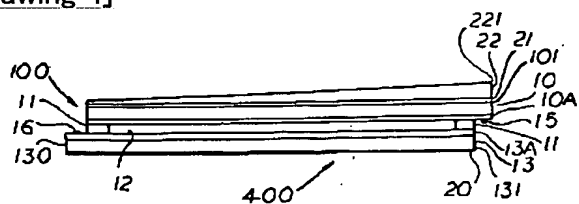
**[Drawing 2]**



**[Drawing 3]**



**[Drawing 4]**



[Translation done.]